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April 17, 2012

SAMPLING AND ANALYSIS PLAN/ QUALITY ASSURANCE PROJECT PLAN OPERABLE UNIT 3, LIBBY ASBESTOS SUPERFUND SITE

Phase V, Part A: Kootenai River Surface Water, Sediment, and Activity-based Sampling

Prepared for and with oversight by:

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SAP/QAPP for OU3, Libby Asbestos Superfund Site
Phase V, Part A: Kootenai River
Surface Water, Sediment, and Activity-Based Sampling

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The Phase V Part A SAP/QAPP for Operable Unit 3 of the Libby Asbestos Superfund Site is approved for implementation without conditions.

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***The most recent versions of field SOPs, FSDS forms, and COC forms are provided electronically in the OU3 eRoom (https://team.cdm.com/eRoom/mt/LibbyOU3). The most recent versions of laboratory and data verification SOPs are provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).*

List of Abbreviations and Acronyms

ABS	activity-based sampling
Ago	area of a grid opening
AOC	Administrative Order on Consent
Cair	air concentration
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHISQ	chi-squared
CI	confidence interval
COC	chain-of-custody
Cw	water concentration
DQO	data quality objective
ED	exposure duration
EDD	electronic data deliverable
EDS	energy dispersive spectroscopy
EF	exposure frequency
EFA	effective filter area
EPC	exposure point concentration
EPA	U.S. Environmental Protection Agency
ET	exposure time
F	fibers
f	indirect preparation dilution factor
f/L	fibers per liter
FSDS	field sample data sheet
FTL	field team leader
GPS	global positioning system
GOx	number of grid openings examined
H&S	health and safety
HASP	health and safety plan
HAZWOPER	**Haz**ardous **W**aste **Op**erations and **E**mergency **R**esponse
HDPE	high-density polyethylene
HEPA	high-efficiency particulate air
HQ	hazard quotient
HV	high volume
ID	identification
IDW	investigation derived waste
ISO	International Organization for Standardization
IUR	inhalation unit risk
KDC	Kootenai Development Corporation
L/cc	liters per cubic centimeter

L/min	liters per minute
L	liters
LA	Libby amphibole
LC	laboratory coordinator
LV	low volume
MCE	mixed cellulose ester
MCL	maximum contaminant level
MDEQ	Montana Department of Environmental Quality
MFL	million fibers per liter
mL	milliliter
mm	millimeter
MWH	MWH Americas, Inc.
N	number of asbestos structures counted
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Laboratory Accreditation Program
NYSDOH	New York State Department of Health
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
OU	operable unit
PCM	phase contrast microscopy
PCME	PCM-equivalent
PDF	portable document format
PE	performance evaluation
PLM	polarized light microscopy
PLM-VE	polarized light microscopy visual area estimation
PLM-Grav	polarized light microscopy gravimetric
PPE	personal protective equipment
PRI-ER	Project Resources, Inc. - Environmental Restoration
QA	quality assurance
QAM	quality assurance manager
QAPP	quality assurance project plan
QA/QC	quality assurance/quality control
QATS	Quality Assurance Technical Support
QC	quality control
RBC	risk-based concentration
RfC	reference concentration
RI	remedial investigation
RI/FS	remedial investigation/feasibility study
ROM	record of modification
RPM	remedial project manager
SAP	sampling and analysis plan

s/cc	structures per cubic centimeter
SOP	standard operating procedure
SPF	sample preparation facility
SRM	standard reference material
TAS	target analytical sensitivity
TEM	transmission electron microscopy
TWFc	cancer time weighting factor
TWFnc	non-cancer time weighting factor
µm	microns
USGS	United States Geological Survey
V	volume
%	percent
±	plus or minus
95UCL	95% upper confidence limit

Section 1 Project Overview

1.1 Purpose of this Document

This document contains the elements required for both a sampling and analysis plan (SAP) and quality assurance project plan (QAPP). This SAP/QAPP describes data collection efforts that will be conducted during Phase V Part A of the remedial investigation (RI) for Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site (the Site).

This SAP/QAPP has been developed in basic accordance with the U.S. Environmental Protection Agency (EPA) *Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA 2001) and the *Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4* (EPA 2006). While this SAP/QAPP is organized differently than the recommended structure in the QA/R-5 guidance, all the required QAPP elements are presented. **Table 1-1** provides a cross-reference where information for each QA/R-5 element is located in this SAP/QAPP. This document is organized as follows:

Section 1 – Project Overview

Section 2 – Background and Problem Definition

Section 3 – Data Quality Objectives

Section 4 – Sampling Program

Section 5 – Sample Preparation and Analysis Requirements

Section 6 – Quality Assurance/Quality Control

Section 7 – Data Management

Section 8 – Assessment and Oversight

Section 9 – Data Validation and Usability

Section 10 – References

All cited tables, figures, and appendices are located at the end of this document, or are provided electronically in the Site eRooms.

1.2 Project Management and Organization

Figure 1-1 presents an organizational chart that illustrates the lines of authority and communication between the agencies and contractors for this project. The following sections summarize the entities and individuals that will be responsible for providing project management, SAP/QAPP development, field sampling support, on-site field coordination, laboratory support, data management, and quality assurance for this project.

1.2.1 Project Management

The EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Christina Progess, EPA Region 8. Ms. Progess is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is John Podolinsky. The EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

The EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC) for performance of a remedial investigation/feasibility study (RI/FS) at OU3 of the Libby Asbestos Site. Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP/QAPP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

1.2.2 SAP/QAPP Development

This SAP/QAPP was developed by CDM Federal Programs Corporation (CDM Smith) at the direction of and with oversight by the EPA. Copies of the SAP/QAPP will be distributed by the CDM Smith Project Manager (or their designate), either in hard copy or in electronic format, as indicated in the distribution list. The CDM Smith Project Manager (or their designate) will distribute updated copies each time a SAP/QAPP revision occurs. A copy of the final, signed SAP/QAPP (and any subsequent revisions) will also be posted to the OU3 website[a] and the OU3 eRoom[b].

1.2.3 Field Sampling Support

All field sampling activities described in this SAP/QAPP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accordance with the sampling plans developed by the EPA. W.R.

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[a] http://cbec.srcinc.com/libby/
[b] https://team.cdm.com/eRoom/mt/LibbyOU3

Grace & Co.-Conn. and KDC will be supported in this field work by MWH Americas, Inc. (MWH), and by their subcontractor Chapman Construction, Inc. Individuals responsible for implementation of field sampling activities in this SAP/QAPP are listed below:

- MWH Project Manager: John Garr
- MWH Field Team Leaders: Kaitlin Barklow/Joan Kester/Blake Downey
- MWH Field Data Quality Control Officer: Betty Van Pelt
- MWH Quality Control Officer: Mike DeDen

1.2.4 On-Site Field Coordination

Access to the mine and other areas of OU3 via Rainy Creek Road is currently restricted and is controlled by the EPA. The on-site point of contact for access to the mine is Rob Burton of Project Resources, Inc. - Environmental Restoration (PRI-ER):

Rob.burton@priworld.com
(406) 293-3690

1.2.5 Laboratory Support

All samples collected as part of this investigation will be sent for preparation and/or analysis at laboratories selected and approved by the EPA. The sample preparation facility (SPF) in Troy, MT will be responsible for the preparation of all sediment samples collected as part of this investigation. Laboratories that will be utilized for analysis asbestos samples may include Hygeia Laboratories, Inc. and/or EMSL Analytical, Inc. Remedium Group, Inc. is responsible for laboratory procurement and establishing subcontracting agreements with the selected laboratories for this investigation.

1.2.6 Data Management

Administration of the master database for OU3 will be performed by EPA contractors. The primary database administrator will be Lynn Woodbury (CDM Smith). The database administrator (or their designate) will be responsible for sample tracking, uploading new data, performing data verification and error checks to identify incorrect, inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed. When the OU3 database has been populated, checked, and validated, relevant asbestos data will be transferred into a Libby Asbestos Site database as directed by the EPA for final storage.

1.2.7 Quality Assurance

There is no individual designated as the EPA Quality Assurance Manager for the Libby project. Rather, the Region 8 quality assurance (QA) program has delegated authority to the EPA RPMs. This means that the EPA RPMs have the ability to review and approve governing investigation documents developed by Site contractors. Thus, it is the responsibility of the EPA RPM for OU3, who is independent of the entities planning and obtaining the data, to ensure that this SAP/QAPP has been prepared in accordance with the EPA QA guidelines and requirements. The EPA RPM is also responsible for managing and overseeing all aspects of the quality assurance/quality control (QA/QC) program for OU3. In this regard, the EPA RPM is supported by the EPA Quality Assurance Technical Support (QATS) contractor, Shaw Environmental, Inc. (Shaw). The QATS contractor will evaluate and monitor QA/QC sampling and is responsible for performing annual audits of each analytical laboratory. In addition, HDR Engineering, Inc. has been contracted by the EPA to provide oversight of field sampling and data collection activities.

Section 2 Background and Problem Formulation

2.1 Site Description

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to contain amphibole asbestos that includes several different mineralogical classifications. For the purposes of the EPA investigations at the Libby Asbestos Superfund Site, this mixture is referred to as Libby amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald *et al.* 1986, McDonald *et al.* 2004, Sullivan 2007, Rohs *et al.* 2007), as well as some residents of Libby (Peipins *et al.* 2003). Based on these adverse effects, the EPA listed the Libby Asbestos Site on the National Priorities List in October 2002.

Starting in 2000, the EPA began conducting a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. Given the size and complexity of the Libby Asbestos Site, the EPA designated a number of OUs. This document focuses on investigations at OU3. OU3 includes the property in and around the former vermiculite mine and the forested areas surrounding the mine that have been affected by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine, including ponds, Rainy Creek, Carney Creek, Fleetwood Creek, and the Kootenai River. Rainy Creek Road is also included in OU3.

Figure 2-1 shows the location of the mine and a preliminary study area boundary for OU3. The EPA established the preliminary study area boundary for the purpose of planning and developing the scope of the RI/FS for OU3. This study area boundary may be revised as data are obtained during the RI for OU3 on the nature and extent of environmental contamination associated with releases that may have occurred from the mine site. The final boundary of OU3 will be defined by the final EPA-approved RI/FS.

2.2 Basis for Concern at OU3

The EPA is concerned with environmental contamination in OU3 because the area is used by humans for a variety of recreational and occupational activities, and also because the area is habitat for a wide range of ecological receptors (both aquatic and terrestrial).

2.3 Scope and Strategy of the Remedial Investigation at OU3

As noted above, Respondents W. R. Grace & Co. - Conn. and KDC are performing an RI in OU3 under EPA oversight in order to characterize the nature and extent of environmental contamination and to collect data to allow the EPA to evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

The RI is being performed in several phases. Phase I of the RI was performed in the fall of 2007 in accordance with the *Phase I Sampling and Analysis Plan for Operable Unit 3* (EPA 2007a). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of asbestos and also other non-asbestos contaminants that might have been released to the environment in the past as a consequence of the mining and milling activities at the site.

Phase II of the OU3 RI was performed in the spring, summer, and fall of 2008. Phase II was composed of three parts, as follows:

- Part A (EPA 2008a) focused on the collection of data on the levels of LA and other chemicals of concern in surface water and sediment, as well as site-specific toxicity testing of surface water using rainbow trout.
- Part B (EPA 2008b) focused on the collection of data on LA levels in ambient air samples collected near the mined area, and on the collection of data on LA and other chemicals of potential concern in groundwater.
- Part C (EPA 2008c) focused on the collection of other data needed to support the ecological risk assessment at the site.

Phase III of the RI (EPA 2009) was performed in the spring, summer, and fall of 2009. Phase III included the collection of activity-based air samples during simulated recreational visitor activities in the forested area, as well as the collection of a variety of ecological community and habitat metrics in support of the ecological risk assessment.

Phase IV of the RI was composed of Part A (activity-based air sampling) and Part B (surface water sampling). Part A (EPA 2010a) was performed in the summer and fall of 2010 and focused on the collection of additional activity-based air samples during simulated recreational visitor, residential wood harvesting, forest management, and firefighting activities to support the human health risk assessment. Part B (EPA 2011a) was performed in the spring and summer of 2011 and focused on the collection of additional site surface water data needed to support the ecological risk assessment. Data collection efforts included sampling and analysis of site surface waters to characterize LA concentrations, as well as efforts to better characterize the habitat suitability of site streams for fish.

Phase V Part A (this document) focuses on characterizing the potential nature and extent of asbestos in surface water and sediment in the Kootenai River. In addition, this investigation will also include the collection of activity-based air samples during simulated recreational activities along the Kootenai River at a popular recreational location.

2.4 Summary of Existing Data

2.4.1 Surface Water and Sediment – LA

Surface water and sediment samples were collected from the Kootenai River during the Phase II Part A sampling investigation. Although the original Phase II Part A sampling design included the collection of Kootenai River samples under both high flow and low flow conditions, samples were only collected during low flow conditions due to safety concerns for personnel sampling under high flow conditions. **Figure 2-2** and **Figure 2-3** provide the sampling stations for surface water and sediment, respectively. Results for LA in surface water and sediment are shown in **Table 2-1** and **Table 2-2**, respectively. As shown, the measured LA surface water concentration upstream of the confluence with Rainy Creek was non-detect, while concentrations immediately downstream of the confluence ranged from non-detect to 0.05 million fibers per liter (MFL) for LA structures longer than 10 microns (μm) and from non-detect to 0.10 MFL for all LA structures. Measured sediment LA concentrations were non-detect upstream of the confluence with Rainy Creek and ranged from non-detect to trace immediately downstream of the confluence.

2.4.2 Surface Water and Sediment – Non-Asbestos

No data are available on surface water or sediment concentrations of non-asbestos contaminants in the Kootenai River. However, surface water and sediment samples were collected and analyzed for non-asbestos contaminants from lower Rainy Creek immediately upstream of the confluence with the Kootenai River (station LRC-6) during the Phase I sampling investigation and during the Phase II Part A sampling investigation under both high flow (Round 1) and low flow (Round 2) conditions. Results for non-asbestos contaminants in surface water and sediment samples for LRC-6 are presented in **Table 2-3** and **Table 2-4**, respectively. As shown, the only non-asbestos contaminants that are consistently detected are metals.

Risks to human health were determined to be below a level of concern for non-asbestos contaminants for all exposure populations (EPA 2012a). The weight of evidence evaluation in the ecological risk assessment for non-asbestos contaminants concluded that risks to aquatic receptors in OU3 creeks and ponds from exposure to non-asbestos contaminants were likely to be minimal (EPA 2012b). Thus, it is concluded that risks from non-asbestos contaminants attributable to possible influences from Rainy Creek are also likely to be below a level of concern or minimal in the Kootenai River. Thus, data are not needed for either surface water or sediment on concentrations of non-asbestos contaminants in the Kootenai River.

2.4.3 Surface Water Flow

Surface water flow data were collected at LRC-6 on a weekly basis in 2008 as part of the Phase II Part A sampling investigation and every half an hour in 2011 as part of the Phase IV Part B sampling investigation. Historical surface water flow data are available online for the Kootenai River below the Libby Dam (USGS 2012). Flow data for the Kootenai River and LRC-6 are presented in Panel A and Panel B of **Figure 2-4,** respectively.

2.4.4 Activity-Based Sampling

As noted above, activity-based sampling (ABS) has been performed for several recreational visitor scenarios as part of the Phase III and Phase IV Part A investigations. However, these sampling efforts did not include a recreational scenario along the Kootenai River. Levels of possible exposure of LA to recreational visitors along the Kootenai River have not yet been quantified.

Section 3 Data Quality Objectives

3.1 Overview of the Process

Data quality objectives (DQOs) define the type, quality, quantity, purpose, and intended uses of data to be collected (EPA 2006). The design of a study is closely tied to its DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and the analyses to be performed. In brief, the DQO process typically follows a seven-step procedure, as follows:

1. State the problem that the study is designed to address
2. Identify the decisions to be made with the data obtained
3. Identify the types of data inputs needed to make the decision
4. Define the bounds (in space and time) of the study
5. Define the decision rule which will be used to make decisions
6. Define the acceptable limits on decision errors
7. Optimize the design using information identified in Steps 1-6

Following these seven steps helps ensure that the project plan is carefully thought out and that the data collected will provide sufficient information to support the key decisions which must be made.

3.2 Data Quality Objectives for Surface Water Collection

3.2.1 State the Problem

There are some data on surface water concentrations of LA for the Kootenai River for use in risk management decisions. However, the existing data have several limitations, as discussed below.

First, existing LA surface water data for the Kootenai River were only collected during low flow conditions in August of 2008. As shown in **Figure 3-1**, measured surface water concentrations in Rainy Creek immediately above the confluence with the Kootenai River (at station LRC-6) show that LA concentrations tend to be highest during the peak of the hydrograph in mid-May. Thus, available data for the Kootenai River do not provide sufficient information to characterize concentrations when potential effects from Rainy Creek are likely to be highest.

Another limitation of the LA results from the Phase II Part A investigation is that the samples were not treated with ozone or ultraviolet light prior to filtration. Studies performed by the EPA

(1983) indicate that measurement of asbestos (both chrysotile and amphibole) in water is complicated by the fact that, if the water is not completely sterile, organic matter associated with microbial contamination tends to form. This causes two effects: a) asbestos fibers in the water tend to clump together within the organic matter, leading to a decrease in structure count because most structures within clumps cannot be identified when analyzing filters using microscopy, and b) fibers within clumps of organic matter tend to adhere to the walls of the sample bottles, thus decreasing the reported concentration of asbestos in the water. To address these issues, the EPA refined protocols for the measurement of LA in surface water at OU3 to require ozonation/ultraviolet treatment of all water samples prior to filtration. Because surface water samples collected as part of the Phase II Part A investigation may have been influenced by fibers clumping and adhering to sampling container walls, previously measured LA concentrations in the Kootenai River samples have the potential to be biased low.

Finally, previous sampling efforts for surface water conducted as part of the Phase II Part A investigation were limited to locations in the Kootenai River that were immediately upstream and downstream of the confluence with Rainy Creek. As noted above, detected LA was present in surface water in the Kootenai River immediately downstream of Rainy Creek. A characterization of concentrations of LA in surface water in the Kootenai River at locations further downstream was not performed.

Thus, measured data are needed to allow the EPA to determine if concentrations of LA in Kootenai River surface water are above a level of human health concern. Furthermore, if the levels of LA are above a level of human health concern, data are needed to determine whether the concentrations of LA in the surface water are attributable to the Rainy Creek drainage and for what distance downstream these affects can be seen.

3.2.2 Identify the Goal of the Study

The goal of this investigation is to provide data that can be used to determine if concentrations of LA in surface water in the Kootenai River downstream of the confluence with Rainy Creek are above a level of human health concern. If concentrations of LA are above a level of concern immediately downstream of the confluence with Rainy Creek, an additional goal of the investigation is to provide data that can be used to determine if LA concentrations in Kootenai River water downstream are elevated relative to concentrations upstream of Rainy Creek and, therefore, can be attributed to Rainy Creek. Lastly, if LA concentrations in Kootenai River water are above a level of human health concern immediately downstream of the confluence with Rainy Creek and elevated concentration levels can be attributed to Rainy Creek, the final goal is to provide data that can be used to determine the spatial extent of LA present in the surface water downstream of the confluence with Rainy Creek.

3.2.3 Identify the Types of Data Needed

Surface Water Data

Reliable and representative measurements of LA concentrations in surface water are needed to evaluate the nature and extent of LA concentrations in the Kootenai River.

Target Analyte List

Surface water samples should be analyzed for LA using transmission electron microscopy (TEM). Reported results should include the size attributes (length, width) of each asbestos structure observed, along with the mineral classification (LA, other amphibole, chrysotile). Meeker *et al*. (2003) noted most LA structures from the Libby ore body contain detectable levels of both sodium and potassium, whereas other potential sources of LA may not. Thus, because it is possible that there could be various sources of LA to the Kootenai River, information on the sodium and potassium content of each LA structure observed, as determined by energy dispersive spectroscopy (EDS), should also be recorded.

Flow and Loading Data

One of the most useful types of information for evaluating the relative significance of water-borne releases is loading (the amount of contaminant carried in water per unit of time). Loading is calculated as the product of concentration and flow. Thus, data on surface water flow rates for lower Rainy Creek (LRC-6) and the Kootenai River downstream of the Libby dam are needed to confirm that sampling occurred during a time period of maximum relative stream loading from Rainy Creek to the Kootenai River.

3.2.4 Define the Bounds of the Study

Spatial Bounds

The primary focus of this investigation is the Kootenai River, with sampling to occur along the distance of the river down to the confluence with Libby Creek (see **Figure 2-1**). Sampling will occur both upstream and downstream of the confluence with Rainy Creek to characterize potential effects from Rainy Creek. Because Rainy Creek is expected to be the principal source of LA to the Kootenai River, samples will also be collected in lower Rainy Creek immediately above the confluence with the Kootenai River (LRC-6) to provide information on possible contributions of LA to the Kootenai River. The LRC-6 data will also help to ensure that measured data in 2012 are consistent with levels that had been measured previously (to avoid the possibility that results from 2012 are biased due to between-year variability). **Figure 3-2** presents the surface water sampling stations (shown as yellow symbols) that will be evaluated in the Phase V Part A investigation.

Temporal Bounds

This investigation will take place during the time period when lower Rainy Creek is contributing the greatest loading relative to the Kootenai River. **Table 3-1** presents a comparison of the average monthly flow for LRC-6 and the Kootenai River for 2008 and 2011 (these are the two years for which flow data are available for LRC-6). As seen, the highest ratio of average monthly flow for LRC-6 to the Kootenai River occurs during the months of April and May (shaded in grey in **Table 3-1**). This year to year variability is due to differences in dam releases and the amount of seasonal runoff between years. Because of this variability, surface water sampling should begin in April and continue through the beginning of June so as to be certain to capture the peak loading from lower Rainy Creek to the Kootenai River for 2012. Surface water sampling will also take place during low flow conditions in the Kootenai River. Low flow conditions are expected to begin sometime in July and continue through October.

Sampling of flow at LRC-6 should begin the first week of April and continue until all other field sampling activities have concluded for this investigation (i.e., October).

3.2.5 Define the Analytic Approach

Concentrations of LA in surface water will be used to support risk management decision-making for human receptors. It is anticipated that their primary use will be to evaluate potential human health risk as a result of using the Kootenai River as a primary drinking water source. Currently, there are no effects thresholds available for ecological receptors for evaluation of LA in surface water.

If concentrations of LA in surface water based on structures greater than 10 μm in length[c] are below the maximum contaminant level (MCL) for asbestos of 7 MFL, risks to humans from consuming water from the Kootenai River as a primary drinking source are below a level of regulatory concern (i.e., asbestos levels in the Kootenai River are as good as, or better than, levels in any other drinking water source).

If concentrations of LA in surface water samples collected immediately downstream of the confluence with Rainy Creek are above the MCL, the Poisson ratio test (Nelson 1982) can be used in making statistical comparisons between individual samples to determine if concentrations are statistically different from those collected upstream of Rainy Creek. The

[c] The asbestos MCL is based on structures greater than 10 μm in length, thus reported water concentrations of LA should also be based on structures greater than 10 μm in length.

Poisson ratio test can also be used to make statistical comparisons between stations that are further downstream to establish the spatial extent of elevated LA in surface water.

3.2.6 Define the Acceptable Limits on Decision Errors

In evaluating the results of surface water samples, two types of decision errors are possible:

- A *false negative decision error* occurs when it is decided that levels of LA in the Kootenai River are below the MCL, when in fact they are above the MCL.

- A *false positive decision error* occurs when it is decided that levels of LA in the Kootenai River are above the MCL, when in fact they are below the MCL.

In the case of classifying individual surface waters samples as either being above or below the MCL, decision errors may occur because of measurement error in the analysis of the sample. For example, if the true concentration of a sample were 6.1 MFL (for structures longer than 10 µm), but the measured concentration were 7.5 MFL (for structures longer than 10 µm), that would result in a false positive decision error.

Measurement error in the analysis of asbestos samples arises because the number of asbestos structures observed during an analysis (and hence the calculated concentration value) is a random variable given by the Poisson distribution:

Observed structure count = Poisson(True Concentration * Volume analyzed)

The relative magnitude of the measurement error is highest when the observed structure counts are low, and decreases as the number of observed structures increases. Therefore, it is possible to control both the false negative and false positive error rates by selecting a target analytical sensitivity (TAS) for samples analyzed by TEM that will yield a relatively high number of structure counts when a sample near the MCL is analyzed. This concept is illustrated in **Figure 3-3**.

As shown, as the TAS is lowered from 100,000 to 50,000 to 10,000 L^{-1}, the probability of both false positive and false negative decisions errors decreases. For example, if a sample with a true concentration of 6 MFL were analyzed, it would be declared to be above the MCL (a false positive decision error) about 9 percent (%) of the time if the TAS were 100,000 L^{-1}, about 3% of the time if the TAS were 50,000 L^{-1}, and 0% if the TAS were 10,000 L^{-1}. Conversely, if the true concentration is 8 MFL, the probability of making a false negative decision error (i.e., incorrectly concluding that the measured concentration is below the MCL) is about 12% if the TAS is 100,000 L^{-1}, 5% if the TAS is 50,000 L^{-1}, and 0% if the TAS is 10,000 L^{-1}.

Based on the relatively small reduction in uncertainty between a TAS of 50,000 and 10,000 L^{-1}, for this investigation, water samples will be analyzed by TEM to a TAS of 50,000 L^{-1}. Unless the true concentration is quite near the MCL, this TAS should be sufficient to achieve low error rates (less than 10%) for both false negative and false positive decision errors. In the event that a lower TAS is needed, additional grid openings may be analyzed in the future to further reduce the probability of decision errors.

3.2.7 Optimize the Design

Study design considerations needed to optimize the nature and extent evaluation of surface water in the Kootenai River are provided in Section 4.

3.3 Data Quality Objectives for Activity-Based Sampling

3.3.1 State the Problem

Previous studies in OU3 have demonstrated that historic releases from mining and milling activities at the vermiculite mine have released LA into the environment and into the watersheds that drain the mine area. Humans may be exposed to LA while recreating in creeks and rivers that may contain LA. Of particular concern are those populations who use the Kootenai River for recreation. Data are needed to support quantitative exposure and risk evaluations to determine what actions may be needed to protect potentially exposed populations during these activities. No data are currently available to evaluate potential inhalation exposures of LA to individuals that may recreate on sandbars in the Kootenai River and along the overbank areas (areas where sediment is deposited on the floodplain of the river bank) of the Kootenai River.

3.3.2 Identify the Goal of the Study

The goal of this investigation is to provide sufficient data to allow the EPA to complete an exposure assessment for recreational visitors along the Kootenai River. The EPA will use the exposure information in an evaluation of potential risks to human health. The risk assessment will support decisions about whether or not response actions are needed to protect human health from unacceptable risks from LA in air during recreational activities on the Kootenai River.

3.3.3 Identify the Types of Data Needed

Types of Disturbance Activities

The principal source medium of concern to people recreating along the Kootenai River is sediments that may contain LA. When the sediments are exposed, especially if they become dry, LA may be released from the sediment due to disturbances during recreational activities, resulting in potential inhalation exposures. It is not feasible to evaluate every possible type of disturbance, so ABS should be performed using scenarios that are considered to be realistic and representative examples of recreational disturbances along the Kootenai River. The key types of activities that may result in disturbances of LA from exposed sediments in overbank areas and sandbars include fishing and dragging boats into and out of the river.

Type of Air Samples

Experience at Libby and at other asbestos-contaminated sites has demonstrated that personal air samples (i.e., samples that collect air in the breathing zone of a person) tend to result in higher concentrations of LA than air samples collected by a stationary monitor (EPA 2007b), especially if the person is engaged in an outdoor activity that disturbs an asbestos source material, such as sediment containing LA. Because personal air samples are more representative of breathing zone exposures, this investigation should focus on the collection of personal air samples during ABS. ABS measurements should be obtained by drawing a known volume of air through a filter that is located in the breathing zone of the individual performing the disturbance activity and measuring the number of LA structures that become deposited on the filter surface.

Sediment Samples

Measured data on the levels of LA in sediment in locations of potential exposure of recreational visitors will be useful in determining the representativeness of the selection of the ABS location. In addition, sediment data will be helpful in comparing the level of LA concentrations between recreational areas.

Sediment sampling should occur during low flow conditions in the Kootenai River because this is when the most sediment is exposed. Based on the annual flow data for the Kootenai River (see **Figure 2-4** Panel A), it appears that the low flow period typically begins in early July and continues through October. Samples of sediment should be collected from the depositional areas along the banks and exposed sandbars within the river channel at locations identified to be commonly used by recreational visitors to the Kootenai River. Recreational areas located in other OUs (e.g., OU1 or OU7) should be excluded from evaluation as part of this study.

Target Analyte List

ABS air samples should be analyzed for LA using TEM. Because asbestos toxicity depends on the particle size and mineral type, results should include the size attributes (length, width) of each asbestos structure observed, along with the mineral classification (LA, other amphibole, chrysotile). In addition, because it is possible that there could be various sources of LA present in sediments, information on the sodium and potassium content of each LA structure observed, as determined by EDS, should also be recorded.

Sediment samples should be analyzed for LA by polarized light microscopy (PLM) using the Libby-specific visual area estimation method (PLM-VE) and gravimetric method (PLM-Grav). In addition, visible vermiculite data should be collected at the time of sample collection.

Exposure Parameters for Recreational Visitors

In addition to measurements of LA concentrations in air during various recreational activities along the Kootenai River, data are also needed on the frequency and duration that recreational visitors spend at the Kootenai River. This includes data on the exposure time (hours/day), exposure frequency (days per year), and exposure duration (years) spent as a recreational visitor to the Kootenai River.

Toxicity Values

In order to estimate potential risks to recreational visitors from the Kootenai River, human health toxicity values are needed to evaluate cancer and non-cancer effects from inhalation exposures to LA. The toxicity value used to evaluate cancer risk is the inhalation unit risk (IUR), and the toxicity value used to evaluate non-cancer risks is the reference concentration (RfC). Draft values for an LA-specific IUR and LA-specific RfC have been developed by the EPA and are currently under review (EPA 2011b).

3.3.4 Define the Bounds of the Study

Spatial Bounds

For the purposes of this ABS investigation, the approach that will be taken at OU3 is to collect ABS samples in a recreational area that is representative of a high frequency of exposure. Based on discussions with local anglers and river guides, the sandbar located in the Kootenai River downstream of Rainy Creek (KR-20 in **Figure 3-2**) has been identified as one location that is used frequently by recreational visitors and is located in an area that may be influenced by mine releases. Thus, this location should be selected for evaluation in the ABS investigation.

Sediment samples should be collected from each of the recreational areas identified in **Figure 3-2** (shown as red symbols) and analyzed for LA. (In addition, sediment samples are also being collected from several recreational areas on the Kootenai River downstream of Libby Creek as part of a separate SAP/QAPP [EPA 2012c].) If levels of LA in sediment at other (non-ABS) recreational areas are greater than levels measured at station KR-20, additional ABS investigations at other recreational areas may be warranted in the future.

Temporal Bounds

Because it is expected that the amount of sediment that may be exposed in overbank areas and sandbars will be highest during low flow, ABS and sediment sampling should occur during low flow conditions. As discussed previously, annual flow data for the Kootenai River (see **Figure 2-4** Panel A) show that the low flow period begins in early July and continues through October. This timing is also optimal for ABS because the release of LA from exposed, dry sediment is likely to be highest when environmental conditions are drier (i.e., summer months). To avoid collecting data that are biased low, ABS sampling should not occur during or within 1 day of rainfall (>1/4 inch). Sampling should also not be conducted if water levels in the Kootenai River rise (e.g., due to Libby dam releases or increased flow from tributaries) to a point where the amount of exposed sediment is no longer representative of the exposure area.

The release of LA from sediment into the air is expected to depend on several factors that may tend to vary over time (e.g., sediment moisture content, wind speed, humidity level). Therefore, ABS data should, to the extent practicable, be collected over a sufficient time frame to ensure the data are representative of the long-term mean concentration level.

3.3.5 Define the Analytic Approach

Measured sediment data will be used to compare the concentrations of LA in sediment between recreational areas, as well as to determine the representativeness of the selected ABS location. Because PLM results for sediment are semi-quantitative, it is expected that the evaluation of PLM results in conjunction with visible vermiculite data will require graphical presentations to evaluate potential spatial patterns of LA.

The ABS air results will be used to calculate an exposure point concentration (EPC). The EPC will be the average ABS air concentration[d] measured over both rounds of sampling. The EPC will be combined with assumptions about exposure frequency and duration for each scenario and toxicity factors for LA in a human health risk assessment that will provide a basis for the

[d] Concentrations will be based on phase contrast microscopy-equivalent (PCME) structures (i.e., structures longer than 5 µm, with a width greater than or equal to 0.25 µm, and an aspect ratio of 3:1 or greater).

EPA to determine, in consultation with MDEQ, whether response action is needed within OU3 to protect human health, and if additional ABS sampling of other recreational locations in the future is warranted.

As noted above, the EPA has recently proposed LA-specific toxicity values for use in estimating cancer risks and non-cancer hazard quotients (HQs) from exposures to LA in air. The EPA is currently reviewing the proposed values. When the toxicity values have been finalized for use in risk assessment, basic methods for estimating human health risk from LA in air will be followed, as specified in the LA-specific Addendum to the *Framework for Investigating Asbestos-Contaminated Superfund Sites* (EPA 2011c.

Decision Rule

EPA guidance contained in Office of Solid Waste and Emergency Response (OSWER) Directive 9355.0-30, Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions (EPA 1991) indicates that if the cumulative cancer risk to an individual based on reasonable maximum exposure for both current and future land use is less than 1E-04, and the non-cancer hazard quotient (HQ) is less than or equal to 1, then remedial action is generally not warranted unless there are adverse environmental impacts. If cancer risk exceeds 1E-04 and/or the HQ exceeds 1, then a response action is generally required. The guidance also states that a risk manager may decide that a risk level lower than 1E-04 is unacceptable and that remedial action may be warranted where there are significant uncertainties in the risk assessment results.

3.3.6 Define the Acceptable Limits on Decision Errors

In making decisions about the risks to humans from exposures to LA in OU3, two types of decision errors are possible:

- A *false negative decision error* would occur if a risk manager decides that exposure to LA in OU3 is not of health concern, when in fact it is of concern.

- A *false positive decision error* would occur if a risk manager decides that exposure to LA in OU3 is above a level of concern, when in fact it is not.

The EPA is most concerned about guarding against the occurrence of false negative decision errors, since an error of this type may leave humans exposed to unacceptable levels of LA in OU3. To minimize the chances of underestimating the true amount of exposure and risk, the EPA generally recommends that risk calculations be based on the 95% upper confidence limit (95UCL) of the sample mean (EPA 1992). Use of the 95% UCL in risk calculations limits the probability of a false negative decision error to no more than 5%. To support this approach, the EPA has developed a software application (ProUCL) to assist with the calculation of 95UCL values (EPA 2010b). However, the equations and functions in ProUCL are not designed for asbestos data sets and application of ProUCL to asbestos data sets is not recommended (EPA 2008d). The EPA is presently working to develop a new software application that will be appropriate for use with asbestos data sets, but the application is not yet available for use. Because the 95UCL cannot presently be calculated with confidence, risk calculations will be based on the sample mean only, as recommended by the EPA (2008d). This means that risk estimates may be either higher or lower than true values, and this will be identified as a source of uncertainty in the risk assessment.

The EPA is also concerned with the probability of making false positive decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. Because it is not possible at present to quantify the uncertainty in the mean of an asbestos data set as a function of the number of samples, it is not possible to specify a minimum number of samples required to minimize the risk of false positive decision errors. However, experience in other outdoor ABS studies at the Libby site indicates that high variability between samples has the potential to occur. Because uncertainty in the sample mean is increased by high variability, the goal is to collect a minimum of four samples at the ABS location. This number of samples should provide a reasonable estimate of average exposure conditions, and provide a reliable basis for calculating the long-term average exposure concentration across multiple sampling events.

3.3.7 Optimize the Design

Study design considerations needed to optimize the ABS sampling for the Kootenai River are provided in Section 4.

Section 4 Sampling Program

All water, sediment, and ABS air data collection activities within OU3 described in this SAP/QAPP will be performed by personnel who are properly trained in the field methods and the experimental sampling design details presented below. The field sampling teams will follow procedures in the OU3-specific Health and Safety Plan (HASP) prepared by MWH for this investigation.

Table 4-1 provides an overview of the number and types of data collection activities that will be performed under Phase V Part A of the OU3 RI. The following sections present the experimental design, including sampling details and rationale, for the Phase V Part A surface water, sediment, and ABS investigations.

4.1 Surface Water Sampling Study Design

This section describes the study design for Phase V Part A data collection activities developed to meet data needs for surface water sampling of the Kootenai River.

4.1.1 Sampling Locations

Figure 3-2 provides a map that shows the locations for the collection of surface water samples (shown as yellow symbols).

4.1.2 Sampling Frequency

High Flow (April-June)

As noted previously, the relative loading from Rainy Creek to the Kootenai River can be variable and depends on multiple factors. In order to ensure that sample collection occurs during the time when Rainy Creek is contributing the greatest relative loading to the Kootenai River, grab samples will be collected from the bank of the river at stations KR-1 and KR-4 during an eight-week time period beginning the week of April 23rd at a frequency of one sample per week. This eight-week time window is expected to span the timeframe of high flow conditions in Rainy Creek and the period of highest influence to the Kootenai River. If flow levels are observed to be at 50% of the peak flow measured in Rainy Creek during week 6, the number of sampling events may be reduced (with week 7 being the final week) and a field modification will be written to document this change. During this eight-week time period grab samples will also be collected from the bank of the river at LRC-6 and from a location in the Kootenai River that is immediately upstream of the confluence with Rainy Creek (UKR-0) at a frequency of one sample per week. Data from LRC-6 and UKR-0 will be used to evaluate the impact of lower Rainy Creek on the Kootenai River.

During the last sampling event in June, one surface water grab sample will be collected from the bank of the river for all stations on the Kootenai River (see **Figure 3-2**) in order to characterize the LA levels during high flow conditions of the Kootenai River. Bank samples will be collected from the northern river bank, barring any river access impediments. In addition, four surface water transect samples (at equally-spaced intervals across the river) will be collected from KR-14 and K-16 on the same day that the grab sample is collected from the bank at these locations. Data collected using the transect approach will be used to compare data from bank-collected grab samples to evaluate the representativeness of the grab samples collected from the bank of the river under high flow conditions.

Table 4-2 presents the surface water sampling frequency for each station that will be evaluated as part of the high flow investigation.

Low Flow (September)

As seen in Panel A of **Figure 2-4**, low flow sampling will likely occur during the month of September. One surface water grab sample will be collected from the bank of the river from all sampling stations (see **Figure 3-2**) during low flow conditions. In addition, four surface water transect samples (at equally-spaced intervals across the river) will be collected from KR-14 and K-16 on the same day that the grab sample is collected from the bank at these locations. Data collected using the transect approach will be used to compare data from bank-collected grab samples to evaluate the representativeness of the grab samples collected from the bank of the river under low flow conditions.

Table 4-2 presents the surface water sampling frequency for each station that will be evaluated as part of the low flow investigation.

4.1.3 Study Variables

It is expected that asbestos concentrations in the Kootenai River are influenced by flow variations and variations in loading from tributaries. The Phase V Part A data should provide information on the range of variability in water concentrations of asbestos as a function of flow fluctuations and loading from tributaries.

4.1.4 Critical Measurements

The critical measurements for this project are measurements of the concentration of LA in surface water. The analysis of LA may be achieved using several different types of microscopes, but the EPA generally recommends using TEM because this analytical method has the ability to clearly distinguish asbestos from non-asbestos structures, and to classify different types of asbestos (i.e., LA, chrysotile).

To ensure that measured concentration data are representative of relative high loading conditions of lower Rainy Creek to the Kootenai River, continuous flow monitoring will be performed at LRC-6 beginning in April and will end when field sampling efforts for this investigation have concluded (i.e., October). Flow data at this station should be recorded using a data logger that is capable of recording water flow measurements at least 1-hour intervals and storing at least one month's worth of measurements. Data should be downloaded and posted to the Libby OU3 eRoom on a weekly basis by MWH. Information regarding the flow of the Kootenai River at station 12301933 will be downloaded from the United States Geological Survey (USGS) National Water Information System web interface (USGS 2012) on a weekly basis by the OU3 data manager (CDM Smith).

4.1.5 Data Reduction and Interpretation

High Flow Surface Water Samples

Water samples collected in the Kootenai River from April to June under high flow conditions (i.e., one sample per week for 8 weeks at stations KR-1 and KR-4) will be filtered by the analytical laboratory and the resulting filters will be used to prepare grids for initial examination by rapid turn-around analysis by TEM (see Section 5). From the rapid turn-around TEM examination, the total number of LA fibers observed is determined and the water concentration is calculated as follows:

$$Cw = (N \cdot EFA) / (GOx \cdot Ago \cdot V \cdot 1E{+}06)$$

where:

Cw	= Water concentration (MFL)
N	= Number of asbestos structures observed (fibers)
EFA	= Effective filter area (mm^2)
GOx	= Number of grid openings examined
Ago	= Area of a grid opening (mm^2)
V	= Volume of water applied to the filter (L)
1E+06	= Conversion factor (f/L --> MFL)

The results of the rapid turn-around TEM analyses will be used to identify a three-week time period when concentrations of LA in the Kootenai River are the highest. All samples collected within this three-week time period (including samples from UKR-0 and LRC-6 within the same three-week time period) will be selected for standard TEM analysis (see Section 5). Other samples outside of this three-week time period will be archived for possible future analysis.

Data on LA concentrations in water generated from the Phase V Part A sampling investigation by standard TEM examination will be used to evaluate potential human health risk from consuming water from the Kootenai River as a primary drinking water source by comparing concentration of LA for structures longer than 10 µm to the MCL.

Surface water samples collected from stations downstream of KR-4 will only be analyzed by TEM as part of this study if measured asbestos concentrations at KR-1 or KR-4 exceed the MCL. If analysis of samples collected downstream of KR-4 is not performed as part of this study, these samples will be archived for possible future analysis.

Low Flow Surface Water Samples

Water samples collected under low flow conditions will be filtered by the analytical laboratory and the resulting filters will be used to prepare grids for standard TEM examination (see Section 5). Samples collected from stations KR-1 and KR-4 will be analyzed first. Surface water samples collected from stations downstream of KR-4 will only be analyzed by TEM as part of this study if measured asbestos concentrations at KR-1 or KR-4 exceed the MCL. If analysis of samples collected downstream of KR-4 is not performed as part of this study, these samples will be archived for possible future analysis.

4.2 Sediment Sampling Study Design

4.2.1 Sampling Locations

Sediment sampling will occur at locations that have been identified as areas that are frequently utilized by recreational visitors and are larger in size. **Figure 3-2** provides a map that shows the stations for the collection of sediment samples (shown as red symbols). In the event that a sample station does not have an adequate amount of sediment that is exposed due to rocky substrate conditions, sediment sampling should not occur at the sampling station (this should be noted in the field logbook).

4.2.2 Sampling Frequency

For the ABS area (KR-20), sediment sampling will be performed prior to the start of each ABS event (i.e., once in August and once in September). For the other sediment stations (see **Figure 3-2**), sediment sampling will consist of one round of sampling to occur during low flow conditions. Annual flow data for the Kootenai River (see **Figure 2-4**) show that the low flow period begins in begins in early July and continues through October. For ease of implementation, sediment collection will occur in September (concomitant with the low flow surface water sampling event).

4.2.3 Study Variables

Levels of LA in sediment will vary across the area of sediment that is exposed. Because recreational visitors to an area may tend to move across the entire exposed area, the sediment sample must provide an average estimate of the LA concentration across the exposed area. Sediment samples will be collected as multi-point composite samples that encompass the entire area to ensure that the sediment results will account for any spatial variability in LA concentrations.

4.2.4 Critical Measurements

A critical measurement associated with this project is the measurement of the concentration of LA in sediment as determined by the Libby-specific PLM methods. In addition, because in Libby the occurrence of visible vermiculite in soil has been shown to be a reliable indicator of the presence of LA in soil (EPA 2010c), field-based estimates of the level of visible vermiculite in sediment are also needed.

4.2.5 Data Reduction and Interpretation

Data for asbestos levels in sediment samples collected as part of the Phase V Part A sampling investigation will be used to provide a frame of reference for the selected recreational area evaluated in the ABS investigation (i.e., KR-20). If levels of LA in sediment at other (non-ABS) recreational areas are greater than levels measured at station KR-20, additional ABS investigations at other recreational areas may be warranted in the future.

4.3 Activity-Based Sampling Study Design

4.3.1 Sampling Location

Multiple locations along the Kootenai River were identified as potential areas for ABS sampling because they are utilized by recreational visitors. Only one location was selected for ABS as part of this Phase V Part A investigation. The location where ABS data will be collected is the sand bar located downstream of the confluence with Rainy Creek (station KR-20 in **Figure 3-2**).

4.3.2 Sampling Frequency

A total of two ABS events will occur at the selected ABS area during low flow conditions. One event will be conducted in August and one event will be conducted in September. For each ABS event, two ABS air samples (one high volume filter and one low volume filter) will be collected for each of two actors. The high volume filter will be preferentially selected for TEM analysis, and the low volume filter will be archived. Thus, a total of four ABS air samples will be analyzed from the ABS area (two events x two actors).

4.3.3 ABS Script

Individuals performing the ABS sampling will engage in a series of scripted activities to generate ABS data that are representative of a range of realistic activities that may be performed by a recreational visitor to the Kootenai River. The script is presented in **Appendix B.** The actors in this script will be simulating an adult person landing a boat, walking around the ABS area and leaving by boat. This script will be representative of typical exposures to fishing guides and recreational anglers.

4.3.4 Study Variables

Because it is recognized that the release of LA from sediment into the air depends on several factors that may tend to vary over time (e.g., sediment moisture content, wind speed, humidity level). ABS air data will, to the extent practicable, be collected over a sufficient time frame to ensure the data are representative of the long-term mean exposure level. Levels of LA in sediment will vary across the area of sediment that is exposed. Because recreational visitors to an area may tend to move across the entire exposed area, the ABS air sample must provide an average estimate of the LA concentration across the exposed area. Thus, scripted activities will be conducted across the entire ABS area.

4.3.5 Critical Measurements

A critical measurement associated with this project is the measurement of the concentration of LA in ABS air. The analysis of LA may be achieved using several different types of microscopes, but the EPA generally recommends using TEM because this analysis method has the ability to clearly distinguish asbestos from non-asbestos structures, and to classify different types of asbestos (i.e., LA, chrysotile). In addition, analysis by TEM provides structure-specific dimensions that allow for the estimation of PCM-equivalent (PCME)[e] concentrations, which is the concentration metric necessary to estimate exposure and risks.

[e] PCME structures have a length greater than 5 µm, width greater than or equal to 0.25 µm, and aspect ratio greater than or equal to 3:1.

4.3.6 Data Reduction and Interpretation

ABS air samples collected in the field will be used to prepare grids for TEM examination (see Section 5). From this examination, the total number of asbestos structures for each type of asbestos is determined and the ABS air concentration is calculated as follows:

$$C_{air} = (N \cdot EFA) / (GOx \cdot Ago \cdot V \cdot 1000 \cdot f)$$

where:

Cair	= Air concentration (structures per cubic centimeter [s/cc])
N	= Number of asbestos PCME structures observed (structures)
EFA	= Effective filter area (mm^2)
GOx	= Number of grid openings examined
Ago	= Area of a grid opening (mm^2)
V	= Sample air volume (L)
1000	= L/cc (conversion factor in liters per cubic centimeter)
f	= Indirect preparation dilution factor (assumed to be 1 for direct preparation)

Data for asbestos concentrations in ABS Air generated from the Phase V Part A sampling investigation will be used to evaluate potential human health risk from recreational exposures along the Kootenai River.

4.4 Sample Collection Methods

4.4.1 Water Sample Collection

As noted in Section 4.1.2, water samples will be collected using two methods, a grab sample collected from the bank of the river and four transect samples collected using a flat-bottom powerboat. All bank-collected water samples will be collected using the direct sampling method described in OU3-specific standard operating procedure (SOP) No. 3, *Surface Water Sampling* (see **Appendix A)** , with the following investigation-specific modifications:

- Measurement of water quality parameters (e.g., pH, specific conductance, etc.) and stream discharge is not required.
- No field filtration of samples will be performed.

- Because dedicated sampling equipment is used for each collected water sample, no equipment rinsates are required.

All transect water samples will be collected in accordance with the collection procedures to be detailed in a new surface water transect sampling SOP, which will be created upon the completion of a peristaltic pump pilot study (see **Appendix H**). This pilot study is designed to ensure that use of the peristaltic pump during transect sampling will not influence fiber concentrations in the water. The surface water transect sampling SOP will be included in this SAP/QAPP as a modification prior to the transect sampling.

Approximately 200-400 milliliters (mL) of water will be collected for each sample and placed into a 500-mL capacity high-density polyethylene (HDPE) wide-mouth, or equivalent, container. Headspace must be left in the collection container to allow for the ozonation/ultraviolet treatment and sonication of the sample by the analytical laboratory prior to analysis. To minimize impacts of field collection activities to subsequent locations downstream, water samples will be collected from downstream to upstream. In addition, if both sediment and water samples will be collected near the same location, water samples will be collected first.

Flow monitoring data for station LRC-6 will be collected in accordance with OU3-specific SOP No. 14, *Installation, Operation, and Maintenance of the Automated Water Sampling and Flow Monitoring Devices* (see **Appendix A)**.

4.4.2 ABS Air Sample Collection

All ABS air samples will be collected in accord with SOP ABS-LIBBY-OU3 (see **Appendix A)**. All air samples will be collected using cassettes that contain a 25 mm diameter mixed cellulose ester (MCE) filter with a pore size of 0.8 µm.

A battery-powered air sampling pump (SKC model AirChek XR5000™ (0.005-5.0 L/min) or similar) will be worn by the participant. The monitoring cassette will be attached to the pump via a plastic tube, and affixed to the shoulder of the participant such that the cassette is within the breathing zone. The breathing zone can be visualized as a hemisphere approximately 6 to 9 inches around an individual's face. The top cover from the cowl extension on the sampling cassette shall be removed ("open-face") and the cassette oriented face down.

Each air sampling pump will be calibrated at the start of each ABS sampling period using the primary calibrator (BIOS Drycal). For pre-sampling purposes, calibration will be considered complete when the measured flow is within ±5% of the target flow, as determined by the mean of three measurements. Each BIOS Drycal used for field calibration will be transported to and from each sampling location in a sealed zip-top plastic bag.

As noted in the ABS script (see **Appendix B**), the pumps should be turned on at the beginning of the ABS event and should be left to run for the duration of the script. Because flow could change during the course of the ABS script, flow will be measured and recorded at the completion of the script. If at any time the observed flow rates are ±10% of the target rate, the sampling pump should be re-calibrated, if possible. If at any time an air sampling pump is found to have faulted or the observed flow rates are 25% below (due to heavy particulate loading or a pump malfunction) or 50% above the target rate, the pump will be replaced or the activity will be terminated. **Figure 4-1** should be consulted to determine the appropriate action. The time elapsed from the start of the activity until the fault/flow observation will be used to determine the appropriate action according to **Figure 4-1**.

Two key variables that may be adjusted during collection of air samples are sampling duration and pump flow rate. The product of these two variables determines the amount of air drawn through the filter, which in turn is an important factor in the analytical cost and feasibility of achieving the target analytical sensitivity (see below). In general, longer sampling times are preferred over shorter sampling times because: a) longer time intervals are more likely to yield representative measures of the average concentration (as opposed to short-term fluctuations); and b) longer collection times are associated with higher volumes, which reduces the number of grid openings that need to be examined to achieve the target analytical sensitivity. Likewise, higher flow rates are generally preferred over lower flow rates because high flow results in high volumes drawn through the filter over shorter sampling times.

ABS personnel should wear two different sampling pumps – a high volume (HV) pump and a low volume (LV) pump. This will allow for the collection of two "replicate" filters (i.e., each filter represents the same sample collection duration, but different total sample air volumes). The appropriate flow rate for each sampling pump should be optimized to achieve the highest sample air volume possible without causing the filter to become overloaded. The flow rate for the high volume pump will be set at 4 L/minute and 2 L/minute for the low volume pump.

4.4.3 Sediment Sample Collection

Sediment samples will be collected, handled, and documented in basic accordance with the procedures specified in OU3-specific SOP No. 1, *Soil Sampling for Non-Volatile Organic Compound Analysis* (see **Appendix A**), with the following investigation-specific modifications:

- It is recognized that this SOP is for soil sampling, but the basic sampling methods are applicable to the collection of exposed sediments.
- Each composite sediment sample will be comprised of 30 individual sampling points that are approximately equidistant from each other and representative of the entire recreational area.

- At each sampling point, collect approximately 50 grams of material. The total mass of sediment material for the composite sample should fill about 1/3 of a gallon-sized zip-top bag.

- The amount of visible vermiculite should be recorded on the field sample data sheet (FSDS) form by field sampling personnel using the principles outlined in SOP CDM-LIBBY-06, *Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties* (see **Appendix A)**. Visible vermiculite will be noted as a presence or absence (number of visible inspection points with vermiculite present and the number of visible inspection points without vermiculite) rather than as the number of points with low, medium, and high amounts of vermiculite in each inspection point as required by SOP CDM-LIBBY-06.

4.5 Global Positioning System Coordinate Collection

If not already collected, the global positioning system (GPS) coordinates will be recorded for each sampling station in basic accordance with the procedures specified in OU3-specific SOP No. 11, *GPS Data Collection* (see **Appendix A**). If necessary, any changes in existing sampling stations should be documented in the field logbook and new GPS coordinates should recorded. If any sampling stations become inaccessible, this information should be documented in the field logbook.

4.6 Equipment Decontamination

Decontamination of non-disposable sampling equipment will be conducted in basic accordance with the procedures specified in OU3-specific SOP No. 7, *Equipment Decontamination* (see **Appendix A**). Materials used in the decontamination process will be disposed of as investigation-derived waste (IDW) as described below.

4.7 Handling Investigation-derived Waste

Any disposable equipment or other IDW will be handled in basic accordance with the procedures specified in OU3-specific SOP No. 12, *IDW Management* (see **Appendix A**).

4.8 Inventory and Procurement of Equipment and Supplies

Prior to initiation of any sampling activities, it is the responsibility of the field team leader (FTL) to review the respective SOPs (see **Appendix A**) and determine the equipment and supplies that are necessary to conduct sampling activities. The FTL will check the field equipment/supply inventory and procure any additional equipment and supplies that are not already contained in the field equipment supply inventory.

The following list summarizes the general equipment and supplies that will be required for most of the studies:

- *Sampling equipment* – See Section 4.4 for sample collection SOPs and medium-specific sampling equipment lists.

- *Field logbook* – Used to document field sampling activities and any problems in sample collection or deviations from this SAP/QAPP. See Section 4.9.1 for standard procedures for field logbooks.

- *Field sample data sheets (FSDSs)* – FSDSs are medium-specific forms that are used to document sample details (i.e., sampling location, Sample number, medium, field QC type, etc.). See Section 4.9.1 for standard procedures for the completion of FSDSs.

- *Sample number labels* – Sample numbers are sequential numbers with investigation-specific prefixes. Sample number labels are pre-printed and checked out to the field teams by the FTL or their designate. To avoid potential transcription errors in the field, multiple labels of the same sample number are prepared – one label is affixed to the collected sample, one label is affixed to the FSDS. Labels may also be affixed to the field logbook or other field documentation forms. See Section 4.9.1 for standard procedures for the completion of FSDSs.

- *Indelible ink pen, permanent marker* – Indelible ink pens are used to complete required manual data entry of information on the FSDS and in the field logbook (pencil may not be used). Permanent markers may be used to write sample numbers on the sample container if pre-printed labels are not available.

- *Personal protective equipment (PPE)* - As required by the HASP.

- *Digital camera* – Used to document sampling locations and conditions.

- *Global positioning system (GPS) unit, measuring wheel, stakes* – Used to identify and mark sampling locations. See Section 4.5 for standard procedures in GPS documentation.

- *Decontamination equipment* – Used to remove any residual asbestos contamination on reusable sampling equipment between the collection of samples. See Section 4.6 for standard decontamination procedures.

4.9 Sample Handling and Custody

4.9.1 Sample Identification and Documentation

Sample Labels

Samples will be labeled with sample identification (ID) numbers supplied by field administrative staff and will be signed out by the sampling teams. Labels for surface water will be affixed to the outside of the sample container and covered with a piece of clear packaging tape. Labels will be affixed on the zip-top sample bag for air samples, and the outside of both the inner and outer zip-top bags for sediment samples.

Sample ID numbers will identify the samples collected during this sampling investigation using the following format:

P5-1####

where:

P5-1 = Prefix that designates samples collected under this Phase V Part A SAP/QAPP
= A sequential four-digit number

Field Documentation

Field teams will record sample information on the most current version of the OU3-specific field sample data sheet (FSDS) for each collected surface water, sediment, and ABS air sample (see **Appendix D**) in accordance with the procedures specified in OU3-specific SOP No. 9, *Field Documentation* (see **Appendix A)**.

The field logbook is an accounting of activities at the Site and will duly note problems or deviations from the governing SAP/QAPP or SOPs. Separate field logbooks will be kept for each study and the cover of each field logbook will clearly indicate the name of the associated study. Field logbooks will be completed prior to leaving a sampling location. Field logbooks will be checked for completeness on a daily basis by the FTL or their designate for the first week of each study. When incorrect field logbook completion procedures are discovered during these checks, the errors will be discussed with the author of the entry and corrected. Erroneous information recorded in a field logbook will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

4.9.2 Field Sample Custody

Field sample custody will follow the requirements specified in OU3-specific SOP No. 9 (see **Appendix A**). In brief, all teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss. All samples and FSDSs will be relinquished by field staff to the field sample coordinator or a designated secure sample storage location at the end of each day.

4.9.3 Chain-of-Custody Requirements

The chain-of-custody (COC) record is employed as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A completed COC record is required to accompany each shipment of samples. Sample custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results by the EPA.

The field sample coordinator will prepare a hard copy COC form using the 3-page carbon copy forms developed specifically for use in this investigation (see **Appendix E**). The bottom copy of the COC will be retained by the field sample coordinator and the other two copies of the COC will accompany the sample shipment.

If any errors are found on a COC after shipment, the hard copy of the COC retained by the field sample coordinator will be corrected and a corrected COC will be provided to the laboratory coordinator (LC) for distribution to the appropriate laboratory.

4.9.4 Sample Packaging and Shipping

Samples will be packaged and shipped in basic accordance with the procedures specified in OU3-specific SOP No. 8, *Sample Handling and Shipping* (see **Appendix A**). In brief, samples will be hand-delivered to the facility or laboratory, picked up by a delivery service courier, or shipped by a delivery service to the designated facility or laboratory, as applicable. For samples requiring shipment, prior to sealing the shipping container, the field sample coordinator will complete the bottom of the COC record and retain the bottom copy of the COC record for the project record. The LC will instruct the field sample coordinator as to the appropriate laboratory for each sample shipment.

4.9.5 Holding Times

In general, there are no holding time requirements for asbestos. Because sample preparation will include techniques to address any issues related to holding time for the medium (e.g., organic material binding asbestos in water), there are no holding time requirements for the surface water, sediment, or ABS air samples collected as part of this sampling investigation.

Section 5 Sample Preparation and Analysis Requirements

5.1 Methods and Requirements

An analytical requirements summary sheet (**OU3VA-0412**), which details the specific analytical and preparation requirements associated with this sampling investigation, is provided in **Appendix G**. A copy of this summary sheet will be submitted with each COC. Libby-specific preparation and analysis SOPs (see **Appendix A**) and Laboratory Modification forms can be found in the Libby Lab eRoom.

5.1.1 Water

Sample Preparation

Water samples selected for analysis should be prepared for asbestos analysis in basic accordance with the techniques in EPA Method 100.2, as modified by Libby Laboratory Modification LB-000020A. In brief, water samples will be prepared using an ozone/ultraviolet treatment that oxidizes organic matter that is present in the water or on the walls of the bottle, destroying the material that causes clumping and binding of asbestos structures. Following treatment, an aliquot of water (generally about 50 milliliters) will be filtered through a 25-mm diameter polycarbonate filter with a pore size of 0.1 µm with a mixed cellulose ester filter (0.45 µm pore size) used as a support filter.

Analysis Methods

For rapid turn-around TEM analyses, approximately one quarter of the filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of ISO 10312:1995(E). Grids will be examined in basic accordance with the procedures described in ISO 10312:1995(E), as modified by an OU3-specific method modification for rapid turn-around analyses TEM (TEM_WATER_Mod1_Rev0). This method modification can be found in **Appendix F**.

For standard TEM analyses, the prepared grids will be examined by TEM in basic accordance with the procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085.

TEM Counting Rules

For TEM analyses, all structures with fibrous morphology, an x-ray diffraction pattern consistent with amphibole asbestos, a energy dispersive spectrum consistent with LA, length greater than or equal to 0.5 µm, and an aspect ratio (length: width) greater than or equal to 3:1

will be counted and recorded. If observed, chrysotile structures will be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

TEM Target Analytical Sensitivity

For TEM analyses, the level of analytical sensitivity needed to ensure that analysis of water samples will be adequate is derived by finding the concentration of LA in water that might be of potential concern, and then ensuring that if a water sample were encountered that had a true concentration equal to that level of concern, it would be quantified with reasonable accuracy. The MCL for asbestos in drinking water is 7 MFL and is based on fibers longer than 10 μm in length. In order to limit false negative and false positive decision errors and keep analytical costs reasonable, the selected TAS for asbestos in water is 50,000 L^{-1} (as discussed in Section 3.2.6).

Maximum Number of LA Structures

Ideally, all samples would be examined by TEM until the target analytical sensitivity is achieved. However, for filters that have high asbestos loading, reliable estimates of concentration may be achieved before achieving the target analytical sensitivity. This is because the uncertainty around a TEM estimate of asbestos concentration in a sample is a function of the number of structures observed during the analysis. The confidence interval (CI) around a count of N structures is characterized as a chi-squared (CHISQ) distribution:

$$N_{true} \sim ½ \cdot CHISQ(2 \cdot N_{observed} + 1)$$

As Nobserved increases, the absolute width of the CI range increases, but the relative uncertainty (expressed as the CI range divided by Nobserved) decreases. This concept is illustrated in **Figure 5-1**. The goal is to specify a target N such that the resulting Poisson variability is not a substantial factor in the evaluation of method precision. As shown in **Figure 5-1**, above about 25 structures, there is little change in the relative uncertainty. Therefore, the count-based stopping rule for TEM should utilize a maximum structure count of 25 LA structures.

Maximum Area to be Examined

The number of grid openings that must be examined (GOx) to achieve the TAS is calculated as:

$$GOx = EFA / (TAS \cdot Ago \cdot V)$$

where:

GOx = Number of grid openings
EFA = Effective filter area (assumed to be 1295 mm^2)
TAS = Target analytical sensitivity $(L)^{-1}$
Ago = Grid opening area (assumed to be 0.01 mm^2)
V = Water volume applied to the filter (L)

Assuming that 0.1 L of water is able to be applied to the filter, a total of 26 grid openings would need to be examined to achieve the TAS. In the event that less water is able to be applied to the filter (due to water turbidity), the number of grid openings that would need to be examined would increase. In order to limit the level of effort (and cost) for any one analysis, the maximum number of grid openings to be examined for this project is 100 grid openings. Assuming that each grid opening has an area of about 0.01 mm^2, this would correspond to a maximum area examined of about 1.0 mm^2.

TEM Stopping Rules

The TEM stopping rules for all water samples from this investigation should be as follows:

1. Count a minimum of two grid openings from each of two grids.
2. Continue counting until one of the following is achieved:
    a. The target analytical sensitivity of 50,000 L^{-1} has been achieved.
    b. 25 LA structures have been observed.
    c. A total filter area of 1.0 mm^2 has been examined (this is approximately 100 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

5.1.2 Sediment

Sample Preparation

All sediment samples collected for asbestos analysis will be transmitted to the sample preparation facility (SPF) located in Troy, MT. Samples will be prepared in accordance with SOP ISSI-LIBBY-01. In brief, the raw sediment sample is dried and then split into two aliquots. One aliquot is placed into archive, and the other aliquot is sieved into coarse (> ¼ inch) and fine fractions. The fine fraction is ground to reduce particles to a diameter of 250 µm or less and this fine-ground portion is split into 4 aliquots.

Sample Analysis

Each sediment sample will be analyzed for LA in accordance with Libby site-specific SOPs. The coarse fraction (if any) will be examined using stereomicroscopy, and any particles of LA will be removed and weighed in accordance with SOP SRC-LIBBY-01, referred to as "PLM-Grav". One of the fine ground fraction aliquots will be analyzed by PLM using the visual area estimation method in accordance with SOP SRC-LIBBY-03, referred to as "PLM-VE". Mass fraction estimates of LA and optical property details will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets.

5.1.3 ABS Air

Because the analytical requirements for the ABS air samples depend upon the LA-specific toxicity values, and these values have not yet been finalized, all collected ABS air samples will be archived for future analysis. Once the LA-specific toxicity values have been finalized, the analytical requirements (i.e., the target analytical sensitivity) will be determined and analyses will be completed.

The analysis of lot blank samples will be completed prior to the collection of any ABS air samples. Collected lot blanks will be prepared and analyzed for LA using TEM in basic accordance with ISO 10312:1995(E) (ISO 1995), with all applicable project-specific laboratory modifications. These modifications include the most recent versions of LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085. A total of 10 grid openings should be examined for each lot blank.

5.2 Data Reporting

Soil Preparation Facility

A local SPF Scribe database is used to track specific information associated with the soil sample preparation process. SPF personnel perform data entry of preparation information from the sample drying and preparation log sheets into an Excel spreadsheet. Preparation data are then uploaded from this spreadsheet into the local SPF Scribe database. Soil sample preparation information will be published to Scribe.NET regularly from the local SPF Scribe project database by the SPF sample coordinator.

Analytical Laboratories

Analytical results will be recorded and results transmitted (including the detailed raw structure data from the TEM analysis) using the OU3-specific EDD spreadsheets for rapid turn-around TEM water results, standard TEM water results, TEM air results, and PLM results[f]. Standard project data reporting requirements will be met for this dataset. Upon completion of the appropriate analyses, EDDs will be posted to the Libby OU3 eRoom within the appropriate turn-around time. Hard copies of all analytical laboratory data packages will be scanned and posted as a portable document format (PDF) to the Libby OU3 eRoom. File names for scanned analytical laboratory data packages will include the laboratory name and the job number to facilitate document organization (e.g., LabX_12345-A.pdf).

5.3 Analytical Turn-around Time

Analytical turn-around time will be negotiated between the LC and the laboratory, with direction from the EPA RPM. It is anticipated that a turn-around times of 2-3 weeks are acceptable for most samples. This may be revised as determined necessary by the EPA.

5.4 Custody Procedures

Soil Preparation Facility

The local SPF Scribe project database is used by the SPF sample coordinator or the ESAT project data manager to prepare an electronic COC. One hard copy of the COC will be generated from the electronic COC and will accompany the sample shipment. The SPF sample coordinator will note the analytical priority level for the samples (based on consultation with the LC) at the top of the COC. The SPF will sign and date the COC and make a copy for the SPF project file. Information on the COC number and analytical laboratory to which the sediment samples were shipped is managed in a spreadsheet maintained by the SPF sample coordinator (or their designate). A copy of this spreadsheet is posted regularly to the Libby Laboratory eRoom.

If any errors are found on a COC after shipment to the analytical laboratory, the hard copy of the COC retained by the SPF sample coordinator will be corrected with a single strikeout, initial, and date. A copy of the corrected COC will be provided to the LC for distribution to the appropriate laboratory. It is the responsibility of the SPF sample coordinator to make any

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[f] The most current version of all EDDs for OU3 are provided in the Libby OU3 eRoom (https://team.cdm.com/eRoom/mt/LibbyOU3).

corrections to the local SPF Scribe project database and publish the corrected data to Scribe.NET.

Analytical Laboratories

Specific laboratory custody procedures are provided in each laboratory's Quality Assurance Management Plan, which have been independently reviewed at the time of laboratory procurement. While specific laboratory sample custody procedures may differ between laboratories, the basic laboratory sample custody process is described briefly below.

Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipment and the individual samples. This inspection will include verifying sample integrity. The accompanying COC record will be cross-referenced with all of the samples in the shipment. The laboratory sample coordinator will sign the COC record, email a copy of the final signed COC to the SPF sample coordinator and the appropriate project data manager, and maintain a copy for their project files.

Depending upon the laboratory-specific tracking procedures, the laboratory sample coordinator may assign a unique laboratory identification number to each sample on the COC. This number, if assigned, will identify the sample through all further handling at the laboratory. It is the responsibility of the laboratory manager to ensure that internal logbooks and records are maintained throughout sample preparation, analysis, and data reporting.

5.5 Archival and Final Disposition

All samples that are prepared at the SPF and are archived will remain at the SPF. All other samples and grids will be maintained in storage at the analytical laboratory unless otherwise directed by the EPA. When authorized by the EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

Section 6 Quality Assurance/Quality Control

6.1 Field

Field quality assurance/quality control (QA/QC) activities include all processes and procedures that have been designed to ensure that field samples are collected and documented properly, and that any issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified. The following sections describe each of the components of the field QA/QC program implemented at the Site.

6.1.1 Field Team Training

Asbestos is a hazardous substance that can increase the risk of cancer and serious non-cancer effects in people who are exposed by inhalation. Therefore, all individuals involved in the collection, packaging, and shipment of samples must have appropriate training. Prior to starting any field work, any new field team member must complete the following, at a minimum:

Training Requirement	Location of Documentation Specifying Training Requirement Completion
Read and understand the governing Health and Safety Plan (HASP)	HASP signature sheet
Attend an orientation session with the field Health and Safety (H&S) manager	Orientation session attendance sheet
Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operations and Emergency Response and relevant 8-hour refreshers	OSHA training certificates
Current 40-hour HAZWOPER medical clearance	Physician letter in the field personnel files
Respiratory protection training, as required by 29 CFR 1910.134	Training certificate
Asbestos awareness training, as required by 29 CFR 1910.1001	Training certificate
Sample collection techniques	Orientation session attendance sheet

It is the responsibility of the field H&S manager to ensure that all training documentation is up-to-date and on-file for each field team member.

A field readiness review meeting will be conducted prior to beginning field sampling activities, to discuss and clarify the following:

- Objectives and scope of the fieldwork
- Equipment and training needs
- Field operating procedures, schedules of events, and individual assignments

- Required QC measures
- Health and safety requirements

It is the responsibility of each field team member to review and understand all applicable governing documents associated with this sampling program, including this SAP/QAPP, all associated SOPs (see **Appendix A**), and the applicable HASP. The FTL will oversee all sample collection activities to ensure that governing documents are implemented appropriately.

6.1.2 Modification Documentation

Minor deviations (i.e., those that will not impact data quality or usability) encountered in day-to-day field work will be noted in the field logbook. Major deviations from this SAP/QAPP that modify the sampling approach and associated guidance documents will be recorded on a field record of modification (ROM) form (see **Appendix C**). Field ROMs will be completed by the FTL, or by assigned field or technical staff. Each completed ROM is assigned a unique number that is specific to each investigation (e.g., Phase V-A LFM-OU3-01) by the EPA RPM or their delegate. Once a form is prepared, it is submitted to the EPA RPM for review and approval. Copies of approved field ROMs are available in the OU3 eRoom and are posted to the OU3 website.

6.1.3 Field Quality Control Samples

Field-based QC samples are those samples which are prepared in the field and submitted to the laboratory in a blind fashion. That is, the laboratory is not aware the sample is a QC sample, and should be treated in the same way as a field sample.

Surface Water

Three types of field QC samples will be collected for surface water as part of this sampling investigation – field blanks, field duplicates, and equipment rinsates.

<u>Field Blank</u>

A field blank is a sample of the same medium as field samples, but which does not contain any contaminant. A field blank for water shall be prepared by placing 400 mL of clean water (e.g., store bought drinking water) into the same type of sample collection container as the field samples. Field blanks will be collected at a frequency of one field blank per week, with one field blank analyzed every other week (submitted for analysis along with the accompanying field samples). It is the responsibility of the FTL to ensure that the appropriate number of field blanks is collected. Field blanks will be given a unique sample number and will be specified as a field blank on the FSDS. The field blanks will be analyzed for asbestos fibers by the same method as will be used for field sample analysis.

If any asbestos structures are observed on a field blank, the FTL and/or laboratory manager will be notified and will take appropriate measures to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "FB" will be added to the related field sample results in the project database to denote that the associated field blank had asbestos structures detected.

Field Duplicate

Field duplicates for water are a second 400-mL water sample collected sequentially from the same station as the parent sample. The field duplicate is collected using the same collection technique as the parent sample. Water field duplicate samples will be collected at a rate of 1 field duplicate per 10 field samples (10%). It is the responsibility of the FTL to ensure that the appropriate number of field duplicates is collected. Each field duplicate is given unique sample number, and field personnel record the sample number of the associated co-located sample in the parent sample number field of the FSDS. The same station location is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as field samples and are blind to the analytical laboratories (i.e., the laboratory cannot distinguish between field samples and field duplicates).

Field duplicate results will be compared to the original parent field sample using the Poisson ratio test using a 90% confidence interval (Nelson 1982). Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability. In general, if more than 20% of field duplicate samples for an investigation are determined to be statistically different, the data usability assessment should alert data users to this inherent variability.

Equipment Rinsates

Equipment rinsates are collected to evaluate potential contamination that arises to due inadequate decontamination of sampling equipment. Equipment rinsates are only required if dedicated sampling equipment is not utilized. For this study, it is anticipated that equipment rinsates will be needed for transect surface water samples, but not bank-collected surface water samples. The collection frequency for equipment rinsate will be one per day. It is the responsibility of the FTL to ensure that the appropriate number of equipment rinsates is collected. Equipment rinsates are samples of water from an uncontaminated source (e.g., store-bought drinking water) that has come into contact with decontaminated sampling equipment. Following decontamination, equipment rinsate water will be placed in the same type of container as used for the field samples (e.g., 500-mL HDPE container). Equipment rinsates will be given a unique sample number and will be specified as an equipment rinsate on the FSDS. The equipment rinsates will be analyzed for asbestos fibers by the same method as will be used for field sample analysis. Equipment rinsates will be blind to the laboratory (i.e., the laboratory will not be able to distinguish between field samples and field blanks).

If any asbestos structures are observed on an equipment rinsate, the FTL and/or laboratory manager will be notified and will take appropriate measures to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "ER" will be added to the related field sample results in the project database to denote that the associated equipment rinsates had asbestos structures detected.

Sediment

Field duplicate samples will be collected as part of the sediment sampling for this investigation. Field duplicates for sediment are collected from the same area as the parent sample but from different individual sampling points. These samples are collected independent of the original field sample with separate sampling equipment and submitted for analysis along with the collected field samples. The field duplicate contains the same number of subsamples as the parent sample (i.e., if the parent sample is a 30-point composite, the field duplicate sample is also a 30-point composite).

Sediment field duplicate samples will be collected at a rate of 1 field duplicate per 10 field samples (10%). It is the responsibility of the FTL to ensure that the appropriate number of field duplicates is collected. Each field duplicate is given a unique sample number, and field personnel record the sample number of the associated co-located sample in the parent sample number field of the FSDS. The same station location is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as field samples and are blind to the laboratories (i.e., the laboratory cannot distinguish between field samples and field duplicates).

Field duplicate results analyzed by PLM will be considered concordant if the reported semi-quantitative bin result for the field duplicate is within one bin of the original parent field sample. The variability between the field duplicate and the associated parent field sample reflects the combined variation in sample heterogeneity and the variation due to measurement error. Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability. In general, if the concordance rate for field duplicate samples is less than 20% for the investigation, the data usability assessment should alert data users to this inherent variability.

ABS Air

Two types of field QC samples will be collected for ABS air as part of this sampling investigation – lot blanks and field blanks for ABS air samples.

<u>Lot Blank</u>

Lot blanks are collected to ensure air samples for asbestos analysis are collected on asbestos-free filters. A lot blank is a randomly selected filter cassette from a manufactured lot. For this sampling effort, two lot blanks will be selected at random from the lot of cassettes to be used for the collection of ABS air samples. It is the responsibility of the FTL to submit the appropriate number of lot blanks prior to cassette use in the field. The lot blanks are analyzed for asbestos by TEM analysis as described above (see Section 5.1.3). Lot blank results will be reviewed by the FTL before any cassette in the lot is used for sample collection. The entire batch of cassettes will be rejected if any asbestos is detected on either lot blank. Only filter lots with acceptable lot blank results are placed into use for the ABS effort.

<u>Field Blank</u>

Field blanks are collected to evaluate potential contamination introduced during sample collection, shipping and handling, or analysis. For this sampling effort, field blanks for ABS air will be collected at a rate of 1 per ABS sampling event. This strategy will generate a total of two field blanks. It is the responsibility of each field team to collect the appropriate number of field blanks. Field blanks are collected by removing the end cap of the sample cassette to expose the filter in the same area where sample collection occurs for about 30 seconds before re-capping the sample cassette. The field blanks are analyzed for asbestos by TEM analysis as described above (see Section 5.1.3).

If any asbestos is observed on a field blank, the FTL and/or laboratory manager will be notified and will take appropriate measures (e.g., re-training on sample collection and analysis procedures) to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "FB" will be added to the related field sample results in the project database to denote that the associated field blank had asbestos structures detected.

6.2 Preparation Facility

All sediment[g] samples submitted for analysis by the Libby-specific PLM methods (i.e., PLM-Grav and PLM-VE) are first processed in accordance with SOP ISSI-LIBBY-01. This processing includes drying, splitting, sieving, grinding, and archiving. These sample processing activities are completed at the sample preparation facility located in Troy, Montana, referred to as the "Troy SPF".

The QA/QC of the soil preparation process is maintained by adherence to standard preparation procedures, submission of preparation QC samples, facilities monitoring, and audits. These procedures and requirements are summarized in below. Detailed information regarding soil preparation procedures and requirements for the Troy SPF can be found in SOP ISSI-LIBBY-01, the *Soil Sample Preparation Work Plan*, and the *ESAT Site Safety Plan*.

6.2.1 Training and Personnel Requirements

Personnel performing sample preparation activities must have read and understood the *Soil Sample Preparation Work Plan*, the *SPF HASP*, and all associated SOPs and governing documents for soil preparation (e.g., SOP ISSI-LIBBY-01). In addition, all personnel must have completed 40-hour OSHA HAZWOPER training, annual updates, annual respirator fit tests, and annual or semi-annual physicals, as required.

Prior to performing activities at the Troy SPF, new personnel will be instructed by an experienced member of the SPF staff and training sessions will be documented in the SPF project files. It is the responsibility of the SPF quality assurance manager (QAM) to ensure that all personnel have completed the required training requirements.

6.2.2 Modification Documentation

When changes or revisions are needed to improve or document specifics about sample preparation procedures used by the Troy SPF, these changes are documented using a laboratory ROM form (see **Appendix C**). The SPF ROM form provides a standardized format for tracking procedural changes in sample preparation and allows project managers to assess potential impacts on the quality of the data being collected. SPF ROMs will be completed by the appropriate SPF or technical staff. Once a form is prepared, it is submitted to the ESAT QAM

[g] For the purposes of this section, the term "soil" will be used to refer to the preparation of all soil and soil-like (e.g., sediment) materials.

(or their designate) for review. Final review and approval is provided by the appropriate EPA RPM. Copies of approved SPF ROMs are available in the Libby Laboratory eRoom.

6.2.3 Preparation QC Samples

Four types of preparation QC samples are collected during the soil preparation process: sand blanks, drying blanks, grinding blanks, and preparation duplicates. Each type of preparation QC sample is described in more detail below.

<u>Sand Blank</u>

A sand blank is a sample of store-bought quartz sand that is analyzed to ensure that the quartz sand matrix used for drying and grinding blanks is asbestos-free. Detailed procedures for this certification process are provided in ESAT SOP PLM-02.00, *Blank Sand Certification by Polarized Light Microscopy*. In brief, for each bag of sand, about 800 grams of sand are removed and split into 40 sand blank aliquots of roughly equal size. Each sand blank is evaluated using stereomicroscopic examination and analyzed by PLM-VE. If a sand blank has detected asbestos, it is re-analyzed by a second PLM analyst to verify the presence of asbestos. The sand is certified as asbestos-free if all 40 sand blanks are non-detect for asbestos. The entire bag of sand is rejected for use if any asbestos is detected in the sand blanks. Only sand bags that are certified as asbestos-free will be utilized in the SPF.

<u>Drying Blank</u>

A drying blank consists of approximately 100 to 200 grams of asbestos-free quartz sand that is processed with each batch of field samples that are dried together (usually this is approximately 125 samples per batch). The drying blank is then processed identically to field samples. Drying blanks determine if cross-contamination between samples is occurring during sample drying. One drying blank will be processed with each drying batch per oven. It is the responsibility of the SPF QAM to ensure that the appropriate number of drying blanks is collected. Each drying blank is given unique sample number that is investigation-specific, as provided by the field sample coordinator (i.e., a subset of sample numbers for each investigation will be provided for use by the SPF). SPF personnel will record the sample number of the drying blank on the sample drying log sheet.

It is the responsibility of the QATS contractor to review the drying blank results and notify the SPF QAM immediately if drying blank results do not meet acceptance criteria and if corrective actions are necessary. If asbestos is detected by PLM-VE in the drying blank (i.e., result is not Bin A), a qualifier of "DB" will be added to the related field sample results in the project database that were dried at the same time as the detected drying blank to denote that the associated drying blank had detected asbestos. In addition, the drying oven will be thoroughly cleaned. If asbestos continues to be detected in drying blanks after cleaning occurs, sample processing must stop and the drying method and decontamination procedures will be evaluated to rectify any cross-contamination issues.

Grinding Blank

A grinding blank consists of asbestos-free quartz sand and is processed along with the field samples on days that field samples are ground. Grinding blanks determine if decontamination procedures of laboratory soil processing equipment used for sample grinding and splitting are adequate to prevent cross-contamination. Grinding blanks are prepared at a frequency of one per grinding batch per grinder per day. It is the responsibility of the SPF QAM to ensure that the appropriate number of grinding blanks are collected. Each grinding blank is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the grinding blank on the sample preparation log sheet.

It is the responsibility of the QATS contractor to review the grinding blank results and notify the SPF QAM immediately if drying blank results do not meet acceptance criteria and if corrective actions are necessary. If any asbestos is detected by PLM-VE in the grinding blank (i.e., result is not Bin A), a qualifier of "GB" will be added to the related field sample results in the project database that were ground at the same time as the detected grinding blank to denote that the associated grinding blank had detected asbestos. In addition, the grinder will be thoroughly cleaned. If asbestos continues to be detected in grinding blanks after cleaning occurs, sample processing must stop and the grinding method and decontamination procedures will be evaluated to rectify any cross-contamination issues.

Preparation Duplicate

Preparation duplicates are splits of field samples submitted for sample preparation. The preparation duplicates are used to evaluate the variability that arises during the soil preparation and analysis steps. After drying, but prior to sieving, a preparation duplicate is prepared by using a riffle splitter to divide the field sample (after an archive split has been created) into two approximately equal portions, creating a parent and duplicate sample.

Preparation duplicate samples are prepared at a rate of 1 per 20 samples (5%) of samples prepared. It is the responsibility of the SPF QAM to ensure that the appropriate number of preparation duplicates is prepared. Each preparation duplicate is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the preparation duplicate and its associated parent field sample on the sample preparation log sheet. Preparation duplicates are submitted blind to the laboratory for analysis by the same analytical method as the parent sample.

Preparation duplicate results will be considered concordant if the reported PLM bin for the preparation duplicate is within one bin of the original parent field sample. The variability between the preparation duplicate and the associated field sample reflects the combined variation due to sample preparation and due to measurement error. Results for preparation duplicate samples are evaluated by the QATS contractor or their designate. If the concordance rate for preparation duplicate samples is less than 10%, the QATS contractor will notify the SPF QAM to determine if corrective action is needed.

6.2.4 Performance Evaluation Standards

The USGS has prepared several Site-specific reference materials of LA in soil that are utilized as performance evaluation (PE) standards to evaluate PLM-VE laboratory accuracy and precision. These PE standards are kept in storage at the Troy SPF and are inserted into the sample train during soil sample processing. In accordance with SOP ISSI-LIBBY-01, PE standards are inserted both pre- and post-processing. PE standards of varying nominal levels will be inserted at a rate of at least one per month per PLM laboratory when soil processing is occurring.

It is the responsibility of the SPF QAM to ensure that the appropriate number of PE standards is inserted. Each PE standard is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the PE standard, the nominal level of the PE standard, and whether it was inserted pre- or post-processing on the sample preparation log sheet. PE standards are submitted blind to the laboratory for analysis by the same analytical method as the field samples.

Results for PE standards will be evaluated by the QATS contractor or their designate. PE standard results are ranked as acceptable if the correct semi-quantitative bin is reported, as determined by the nominal concentration of the PE standard. The LC should be notified if PE standard results do not meet acceptance criteria. Corrective action will be taken if the PE standards demonstrate issues with accuracy and/or bias in PLM-VE results reporting. Examples of corrective actions that may be taken include reanalysis and/or repreparation, collaboration between and among laboratories to address potential differences in analysis methods, and analyst re-training.

6.3 Analytical Laboratory

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific QA program designed to ensure accuracy of analytical and consistency of reported analytical results between laboratories. These laboratories are audited by the EPA QATS contractor (see Section 8.1.2) and the National Voluntary Laboratory Accreditation Program (NVLAP) on a regular basis.

Laboratory QA/QC activities include all processes and procedures that have been designed to ensure that data generated by an analytical laboratory are of high quality and that any problems in sample preparation or analysis that may occur are quickly identified and rectified. Laboratories handling samples collected as part of this sampling investigation will be provided a copy of and will adhere to the requirements of this SAP/QAPP. This section describes the laboratory QA/QC procedures that are required of each laboratory that analyzes field samples from OU3.

6.3.1 Laboratory Quality Assurance Management Plan

Each analytical laboratory has developed a laboratory-specific *QA Management Plan* that provides a detailed description of the procedures and policies that are in place at their laboratory to ensure laboratory quality. This laboratory *QA Management Plan* will include information on standard laboratory methods and SOPs, instrument testing, inspection, maintenance, and calibration requirements, procedures for inspection of supplies and consumables, analyst training, facility contamination monitoring, and internal auditing. These laboratory *QA Management Plans* are reviewed and approved by the LC when the subcontracting agreement is established. Copies of all laboratory *QA Management Plans* for each project laboratory are maintained by the LC. The QATS contractor will also review the laboratory *QA Management Plan* during the annual EPA laboratory audit (see Section 8.1.2 below).

6.3.2 Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Each laboratory is accredited by the National Institute of Standards and Technology (NIST)/NVLAP for the analysis of airborne asbestos by TEM and/or analysis of bulk asbestos by PLM. This includes the analysis of NIST/NVLAP standard reference materials (SRMs), or other verified quantitative standards, and successful participation in two proficiency rounds per year each of bulk asbestos by PLM and airborne asbestos by TEM supplied by NIST/NVLAP.

Copies of recent proficiency examinations from NVLAP or an equivalent program are maintained by each participating analytical laboratory. Many of the laboratories also maintain certifications from other state and local agencies. Copies of all proficiency examinations and certifications are also maintained by the LC.

Each laboratory working on the Libby project is also required to pass an on-site EPA laboratory audit. The details of this EPA audit are discussed in Section 8.1.2. The LC also reserves the right to conduct any additional investigations deemed necessary to determine the ability of each laboratory to perform the work. Each laboratory also maintains appropriate certifications from the state and possibly other certifying bodies (e.g., New York State Department of Health (NYSDOH)) for methods and parameters that may also be of interest to the Libby project. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Laboratory personnel working on the Libby project are reviewed for requisite experience and technical competence to perform asbestos analyses. Copies of personnel resumes are maintained for each participating laboratory by the LC in the Libby project file.

6.3.3 Laboratory Team Training/Mentoring Program

Initial Mentoring

The orientation program to help new laboratories gain the skills needed to perform reliable analyses at the Site involves successful completion of a training/mentoring program that was developed for new laboratories prior to their analysis of Libby field samples. All new laboratories are required to participate in this program. The training program includes a rigorous 2-3 day period of on-site training provided by senior personnel from those laboratories already under contract on the Libby project, with oversight by the QATS contractor. The tutorial process includes a review of morphological, optical, chemical, and electron diffraction characteristics of LA, as well as training on project-specific analytical methodology, documentation, and administrative procedures used on the Libby site. The mentor will also review the analysis of at least one sample by each type of analytical method with the trainee laboratory.

Site-Specific Reference Materials

TEM - Because LA is not a common form of asbestos, USGS prepared site-specific reference materials using LA collected at the Libby mine site (EPA 2008e). Upon entry into the Libby program, each laboratory is provided samples of these LA reference materials. Each laboratory is required to analyze multiple LA structures present in these samples by TEM in order to become familiar with the physical and chemical appearance of LA and to establish a reference library of LA EDS spectra. These laboratory-specific and instrument-specific LA reference spectra (EPA 2008f) serve to guide the classification of asbestos structures observed in Libby field samples during TEM analysis.

PLM - USGS has also prepared site-specific reference materials of LA in soil for use during PLM visual area estimation analysis (EPA 2008f). These reference materials were prepared by adding aliquots of LA spiking material to uncontaminated Libby soils to obtain nominal LA concentrations of 0.2% and 1.0% (by weight). Each laboratory was provided with samples of these reference materials for use in training PLM analysts in the visual area estimation of LA levels in soil. In addition, aliquots of these reference materials (as well as other spiked soils) are also utilized as PE standards to evaluate PLM laboratory accuracy.

Regular Technical Discussions

On-going training and communication is an essential component of QA for the Libby project. To ensure that all laboratories are aware of any technical or procedural issues that may arise, a regular teleconference is held between the EPA, their contractors, and each of the participating laboratories. Other experts (e.g., USGS) are invited to participate when needed. These calls cover all aspects of the analytical process, including sample flow, information processing, technical issues, analytical method procedures and development, documentation issues, project-specific laboratory modifications, and pertinent asbestos publications.

Professional/Technical Meetings

Another important aspect of laboratory team training has been the participation in technical conferences. The first of these technical conferences was hosted by USGS in Denver, Colorado, in February 2001, and was followed by another held in December 2002. The Libby laboratory team has also convened on multiple occasions at the ASTM Johnston Conference in Burlington, Vermont, including in July 2002, July 2005, July 2008, and July 2011, and at the Michael E. Beard Asbestos Conference in San Antonio, Texas in January 2010. In addition, members of the Libby laboratory team attended an EPA workshop to develop a method to determine whether LA is present in a sample of vermiculite attic insulation held in February 2004 in Alexandria, Virginia. These conferences enable the Libby laboratory and technical team members to have an on-going exchange of information regarding all analytical and technical aspects of the project, including the benefits of learning about developments by others.

6.3.4 Analyst Training

TEM

All TEM analysts for the Libby project undergo extensive training to understand TEM theory and the application of standard laboratory procedures and methodologies. The training is typically performed by a combination of personnel, including the laboratory manager, the laboratory QAM, and senior TEM analysts.

In addition to the standard TEM training requirements, trainees involved with the Libby project must familiarize themselves with Site-specific method deviations, project-specific documents, and visual references. Standard samples that are often used during TEM training include known pure (traceable) samples of chrysotile, amosite, crocidolite, tremolite, actinolite and anthophyllite, as well as fibrous non-asbestos minerals such as vermiculite, gypsum, antigorite, kaolinite, and sepiolite. New TEM analysts on the Libby project are also required to perform an *EDS Spectra Characterization Study* (EPA 2008f) on the LA-specific reference materials provided during the initial training program to aide in LA mineralogy recognition and definition. Satisfactory completion of each of these tasks must be approved by a senior TEM analyst.

All TEM analysts are also trained in the Site-specific laboratory QA/QC program requirements for TEM. The entire program is discussed to ensure understanding of requirements and responsibilities. In addition, analysts are trained in the project-specific reporting requirements and data reporting tools utilized in transmitting results. Upon completion of training, the TEM analyst is enrolled as an active participant in the Libby laboratory program.

A training checklist or logbook is used to assure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all TEM analysts have completed the required training requirements.

<u>PLM</u>

All PLM analysts for the Libby project are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, National Institute of Occupational Safety and Health (NIOSH) Method 9002, CARB Method 435, and Site-specific SOPs SRC-LIBBY-01 and SRC-LIBBY-03. Analysts with less than one year of experience specific to the Libby project are required to participate in the laboratory mentoring program to obtain additional guidance and instruction. This training is provided by the laboratory managers and/or senior PLM analysts that are familiar with the types of asbestos and analytical challenges encountered at the Site. Before performing any Site analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision for the LA-specific reference materials.

Satisfactory completion of each of these training tasks must be approved by a senior PLM analyst. A training checklist or logbook is used to ensure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all analysts have completed the required training requirements.

6.3.5 Modification Documentation

When changes or revisions are needed to improve or document specifics about analytical methods or procedures used by the laboratory, these changes are documented using a laboratory ROM form (see **Appendix C**). The laboratory ROM form provides a standardized format for tracking procedural changes in sample analysis and allows project managers to assess potential impacts on the quality of the data being collected. Laboratory ROMs will be completed by the appropriate laboratory or technical staff. Once a form is prepared, it is submitted to the EPA RPM for review and approval. Copies of approved laboratory ROMs are available in the OU3 eRoom.

6.3.6 Analytical Laboratory QC Analyses

TEM

The Libby-specific QC requirements for TEM analyses of asbestos are patterned after the requirements set forth by National Voluntary Laboratory Accreditation Program (NVLAP). In brief, there are three types of laboratory-based QC analyses that are performed for TEM – laboratory blanks, recounts, and repreparations. Detailed information on the Libby-specific requirements for each type of TEM QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in the most recent version of Libby Laboratory Modification LB-000029, with the following investigation-specific modifications:

- Laboratory QC sample frequency requirements should be applied on an OU3-specific and medium-specific basis, rather than "across all media" as specified in LB-000029.

- Inter-laboratory analyses should be performed at a minimum frequency of 2% and repreparations at a minimum frequency of 4%.

With the exception of inter-laboratory analyses, it is the responsibility of the laboratory manager to ensure that the proper number of TEM QC analyses are completed. Inter-laboratory analyses for TEM will selected *post hoc* by the QATS contractor or their designate in accordance with the selection procedures presented in LB-000029. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories.

PLM

Laboratory QC for PLM-Grav is ensured through compliance with laboratory-based QC requirements for the NIOSH Method 9002, as specified by NVLAP. No additional project-specific QC requirements have been established for PLM-Grav.

Laboratory-based QC requirements for PLM-VE are specified in SOP SRC-LIBBY-03. Three types of laboratory-based QC analyses are performed for PLM-VE, including laboratory duplicates, inter-laboratory analyses, and PE standards. Detailed information on the Libby-specific requirements for each type of PLM-VE QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in SOP SRC-LIBBY-03, with the following investigation-specific modifications:

- Laboratory QC sample frequency requirements should be applied on an OU3-specific basis.

With the exception of inter-laboratory analyses, it is the responsibility of the laboratory manager to ensure that the proper number of PLM-VE QC analyses are completed. Inter-laboratory analyses for PLM-VE will selected *post hoc* by the QATS contractor or their designate in accordance with the selection procedures presented in SOP SRC-LIBBY-03. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories.

6.4 Instrument Maintenance and Calibration

6.4.1 Field Equipment

All field equipment should be maintained and calibrated in basic accordance with manufacturer specifications. When a piece of equipment is found to be operating incorrectly, the piece of equipment will be labeled "out of order" and placed in a separate area from the rest of the sampling equipment. The person who identified the equipment as "out of order" will notify the FTL overseeing the investigation activities. It is the responsibility of the FTL to facilitate repair of the out-of-order equipment. This may include having appropriately trained field team members complete the repair or shipping the malfunctioning equipment to the manufacturer. Field team members will have access to basic tools required to make field acceptable repairs. This will ensure timely repair of any "out of order" equipment.

6.4.2 Sample Preparation Equipment

Sediment processing instrumentation requiring calibration or routine function checks include sample grinders, drying ovens, ventilation hood, high-efficiency particulate air (HEPA) vacuum, hood anemometer, and the analytical balance. A detailed description of the calibration and maintenance procedures for each type of equipment is provided in the *Soil Sample Preparation Work Plan*.

Calibration and maintenance checks are documented on equipment-specific calibration and maintenance log sheets, as provided in SOP ISSI-LIBBY-01, Attachments 4 through 6. These calibration and maintenance log sheets are kept in a ringed binder, pre-numbered with the equipment number and arranged according to equipment type. It is the responsibility of the SPF QAM (or their designate) to verify that the calibration of each piece of equipment is checked daily and is operating within normal parameters.

6.4.3 Laboratory Instruments

The laboratory manager is responsible for ensuring that all laboratory instruments used for this project are maintained and calibrated in accordance with the manufacturer's instructions. If any deficiencies in instrument function are identified, all analyses shall be halted until the deficiency is corrected. The laboratory shall maintain a log that documents all routine maintenance and calibration activities, as well as any significant repair events, including documentation that the deficiency has been corrected.

6.5 Inspection/Acceptance of Supplies and Consumables

6.5.1 Field

In advance of field activities, the FTL will check the field equipment/supply inventory and procure any additional equipment and supplies that are needed. The FTL will also ensure any in-house measurement and test equipment used to collect data/samples as part of this SAP/QAPP is in good, working order, and any procured equipment is acceptance tested prior to use. Any items that the FTL determines unacceptable will be removed from inventory and repaired or replaced as necessary.

6.5.2 SPF and Analytical Laboratory

The SPF and laboratory managers are responsible for ensuring that all reagents and disposable equipment used in this project are free of asbestos contamination. This is demonstrated by the collection of blank samples.

Section 7 Data Management

All data generated as part of the Phase V Part A sampling investigation will be maintained in an OU3-specific Microsoft Access® database. This will be a relational database with tables designed to store information on station location, sample collection details, preparation and analysis details, and analytical results. Results will include all asbestos data, including detailed structure attributes for TEM analyses.

7.1 Roles and Responsibilities for Data Flow

7.1.1 Field Personnel

W. R. Grace & Co.-Conn. contractors will perform all Phase V Part A sample collection in accordance with this SAP/QAPP. In the field, sample details will be documented on hard copy media-specific FSDS forms and in field log books. COC information will be documented on hard copy forms. FSDS and COC information will be manually entered into a field-specific[h] OU3 database using electronic data entry forms. Use of electronic data entry forms ensures the accuracy of data entry and helps maintain data integrity. For example, data entry forms utilize drop-down menus and check boxes whenever possible. These features allow the data entry personnel to select from a set of standard inputs, thereby preventing duplication and transcription errors and limiting the number of available selections (e.g., media types). In addition, entry into a database allows for the incorporation of data entry checks. For example, the database will allow a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

Entry of FSDS forms and COC information will be completed weekly, or more frequently as conditions permit. Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to the OU3 eRoom[i] site on a weekly basis. This eRoom will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20110412.pdf). Electronic copies of all digital photographs will also be posted weekly to the Libby OU3 eRoom. File names for digital

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[h] The field-specific OU3 database is a simplified version of the master OU3 database. This simplified database includes only the station and sample recording and tracking tables, as well as the FSDS and COC data entry forms.
[i] https://team.cdm.com/eRoom/mt/LibbyOU3

photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20110412_12345.tif).

After FSDS data entry is completed, a copy of the field-specific OU3 database will be posted by the field data manager to the Libby OU3 eRoom weekly, or more frequently as conditions permit. The field-specific OU3 database posted to the eRoom site will include the post date in the file name (e.g., FieldOU3DB_20110516.mdb).

Flow data will be downloaded from the data logger and posted to the Libby OU3 eRoom on a weekly basis. File names for flow output files will include the station identifier and the associated date range (e.g., ST-1_2011_0412-0419.tif).

7.1.2 SPF Personnel

The Troy SPF utilizes a local SPF Scribe project database to maintain soil sample preparation information. Soil preparation information from the preparation log sheets is entered into the local SPF Scribe project database by SPF personnel. After the data entry is checked against the original forms, it is the responsibility of the SPF manager (or their designate) to publish soil sample preparation information from the local SPF Scribe database to Scribe.NET.

It is the responsibility of the OU3 data manager (CDM Smith) to subscribe to the SPF Scribe project database and upload relevant information on soil sample preparation (e.g., mass associated with each sample fraction) and COC tracking details for OU3 samples into the master OU3 project database.

7.1.3 Analytical Laboratory Personnel

As described in Section 5.2, each of the laboratories performing asbestos analyses for the Phase V Part A sampling investigation are required to utilize all applicable OU3-specific Microsoft Excel® spreadsheets for asbestos data recording and electronic submittals. Upon completion of the appropriate analyses, EDDs along with scanned copies of all analytical laboratory data packages will be posted to the Libby OU3 eRoom.

7.1.4 Database Administrators

Day-to-day operations of the master OU3 database will be under the control of EPA contractors. The primary database administrator (CDM Smith) will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the master OU3 database within an appropriate time period of FSDS and/or EDD receipt.

7.2 Master OU3 Project Database

The master OU3 project database is a relational Microsoft Access® database developed specifically for OU3. The *Libby OU3 Database User's Guide* provides an overview of the master OU3 project database structure and content. The most recent version of this *User's Guide* is provided on the OU3 website.

The master OU3 project database is kept on the CDM Smith server in Denver, Colorado. Incremental backups of the master OU3 project database are performed daily Monday through Friday, and a full backup is performed each Saturday. The full backup tapes are stored off-site for 30 days. After 30 days, the tape is placed back into the tape library to be overwritten by another full backup.

7.3 Data Reporting

Field summary reports are prepared by Remedium's field collection contractor. Analytical results summaries are included in the OU3 investigation-specific SAPs and will be provided in the Data Summary Report (in preparation), which are available on the OU3 website. Specialized requests for data summaries may be submitted to the EPA RPM.

7.4 Data Storage

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA RPM. At the termination of this project, all original data records will be provided to the EPA RPM for incorporation into the Site project files.

Section 8 Assessment and Oversight

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that any deviations from procedures are documented. These reports also serve to keep management current on field activities.

8.1 Assessments

8.1.1 Field Oversight

The EPA field oversight contractor (HDR Engineering) will perform field audits of sampling collection activities as part of the surface water and ABS air collection efforts. The EPA field auditor has the authority to direct changes in field activities, or to halt field activities if needed until a remedy to an unexpected problem can be identified. Field audit findings are documented in audit reports issued by the entity performing the audit, and are often discussed with the project management team before the auditors leave the Site. Corrective actions will be immediately implemented, as appropriate. A copy of the field audit report will be provided to the EPA RPM and the QATS contractor.

8.1.2 SPF Audits

Internal audits of the SPF are conducted by the SPF QAM periodically to evaluate personnel in their day-to-day activities and to ensure that all processes and procedures are performed in accordance with governing documents and SOPs. All aspects of sample preparation, as well as sample handling, custody, and shipping are evaluated. If any issues are identified, SPF personnel are notified and retrained as appropriate. Audit reports will be completed following each laboratory audit. A copy of the internal audit report, as well as any corrective action reports, will be provided to the LC and the QATS contractor.

Internal audits will be conducted following any significant procedural changes to the soil preparation processes or other SPF governing documents, to ensure the new methods are implemented and followed appropriately.

The Troy SPF is also required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. Audits consist of an evaluation of facility practices and procedures associated with the preparation of soil samples. A checklist of requirements, as derived from the applicable governing documents and SOPs, is prepared by the auditor prior to the audit, and used during the on-site evaluation. Evaluation of the facility is made by reviewing SPF documentation, observing sample processing, and interviewing personnel.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report following the SPF audit. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each SPF to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the on-site audit findings and recommendations. The purpose of this reported is to identify SPF performance problems and isolate the potential causes.

8.1.3 Laboratory Audits

Each laboratory working on the Libby project is required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. These audits are performed by EPA personnel (and their contractors), that are external to and independent of, the Libby laboratory team members. These audits ensure that each analytical laboratory meets the basic capability and quality standards associated with analytical methods for asbestos used at the Libby site. They also provide information on the availability of sufficient laboratory capacity to meet potential testing needs associated with the Site.

<u>External Audits</u>

Audits consist of several days of technical and evidentiary review of each laboratory. The technical portion of the audit involves an evaluation of laboratory practices and procedures associated with the preparation and analysis of samples for the identification of asbestos. The evidentiary portion of the audit involves an evaluation of data packages, record keeping, SOPs, and the laboratory QA manual. A checklist of method-specific requirements for the commonly used methods for asbestos analysis is prepared by the auditor prior to the audit, and used during the on-site laboratory evaluation.

Evaluation of the capability for a laboratory to analyze a sample by a specific method is made by observing analysts performing actual sample analyses and interviewing each analyst responsible for the analyses. Observations and responses to questions concerning items on each method-specific checklist are noted. The determination as to whether the laboratory has the capability to analyze a sample by a specific method depends on how well the analysts follow the protocols detailed in the formal method, how well the analysts follow the laboratory-specific method SOPs, and how the analysts respond to method-specific questions.

Evaluation of the laboratory to be sufficient in the evidentiary aspect of the audit is made by reviewing laboratory documentation and interviewing laboratory personnel responsible for maintaining laboratory documentation. This includes personnel responsible for sample check-in, data review, QA procedures, document control, and record archiving. Certain analysts responsible for method quality control, instrument calibration, and document control are also interviewed in this aspect of the audit. Determination as to the capability to be sufficient in this aspect is made based on staff responses to questions and a review of archived data packages and QC documents.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report for each analytical laboratory participating in the Libby program. These reports are handled as business confidential items. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each laboratory to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the on-site audit findings and recommendations. The purpose of this reported is to identify common asbestos laboratory performance problems and isolate the potential causes.

<u>Internal Audits</u>

Each laboratory will also conduct periodic internal audits of their specific operations. Details on these internal audits are provided in the laboratory *QA Management Plan*. The laboratory QAM should immediately contact the LC and the QATS contractor if any issues are identified during internal audits that may impact data quality for OU3 samples.

8.2 Response Actions

Corrective response actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken to immediately correct a quality problem will be documented in the applicable field or laboratory logbooks and a verbal report will be provided to the appropriate manager (e.g., the FTL or LC). Major corrective actions will be approved by the EPA RPM and the appropriate manager prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. EPA RPM for OU3 will be notified when quality problems arise that that cannot be corrected quickly through routine procedures (contact information is provided below):

Christina Progess
U.S. EPA Region 8
1595 Wynkoop Street
Denver, CO 80202
Tel: (303) 312-6009
Fax: (303) 312-7151
E-mail: progess.christina@epa.gov

In addition, when modifications to this SAP/QAPP are required, either for field or laboratory activities, a ROM must be completed and approved by the EPA RPM prior to implementation.

8.3 Reports to Management

No regularly-scheduled written reports to management are planned as part of this project. However, reports will be provided to management for routine audits and whenever quality problems are encountered. Field and analytical staff will promptly communicate any difficulties or problems in implementation of the SAP/QAPP to the EPA, and may recommend changes as needed. If any revisions to this SAP/QAPP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

Section 9 Data Validation and Usability

9.1 Data Review, Verification and Validation

9.1.1 Data Review

Data review of project data typically occurs at the time of data reporting by the data users and includes cross-checking that sample IDs and sample dates have been reported correctly and that calculated analytical sensitivities or reported values are as expected. If discrepancies are found, the data user will contact the database administrator (CDM Smith), who will then notify the appropriate entity (field, preparation facility, or laboratory) in order to correct the issue.

9.1.2 Criteria for LA Measurement Acceptability

Several factors are considered in determining the acceptability of LA measurements in surface water and ABS air samples analyzed by TEM. This includes the following:

- *Evenness of filter loading.* This is evaluated using a CHISQ test, as described in ISO 10312 Annex E. If a filter fails the chi-square test for evenness, the result may not be representative of the true concentration in the sample, and the results should be given low confidence.

- *Results of QC samples.* This includes both field and laboratory QC samples, such as field and laboratory blank samples, field duplicates, and various types of recount and re-preparation analyses. If significant LA contamination is detected in field or laboratory blanks, all samples prepared on that day should be considered to be potentially biased high. If agreement between original analyses and field or laboratory duplicates (i.e., repreparations, recount analyses) is poor, results for those samples should be given low confidence.

For PLM analyses, the following factors will be considered in determining the acceptability of LA measurements sediment samples:

- *Results of performance evaluation (PE) standard analyses.* PLM accuracy of visual area estimation results is evaluated using LA-specific PE standards. If the results for these PE standards are not within the project-specific acceptance criteria, results should be given low confidence.

- *Results of QC samples.* This includes field, preparation, and laboratory QC samples. If agreement between original and repeat analyses (i.e., duplicate analyses, inter-laboratory analyses) is strongly discordant, results for those samples should be given low confidence. If significant LA contamination is detected in preparation blanks, all samples prepared on that day should be considered to be potentially biased high.

9.1.3 Data Verification Method

Data verification includes checking that results have been transferred correctly from the original hand-written, hard copy field and analytical laboratory documentation to the OU3 project database. The goal of data verification is to identify and correct data reporting errors.

For analytical laboratories that utilize the OU3-specific EDD spreadsheets, data checking of reported analytical results begins with automatic QC checks that have been built into the spreadsheets. In addition to these automated checks, a detailed manual data verification effort will be performed for 10% of all surface water and sediment samples and analysis results. This data verification process utilizes Site-specific SOPs developed to ensure TEM and PLM results and field sample information in the OU3 database are accurate and reliable:

- EPA-LIBBY-09 – SOP for TEM Data Review and Data Entry Verification – This Site-specific SOP describes the steps for the verification of TEM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-10 - SOP for PLM Data Review and Data Entry Verification – This Site-specific SOP describes the steps for the verification of PLM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-11- SOP for FSDS Data Review and Data Entry Verification – This Site-specific SOP describes the steps for the verification of field sample information, based on a review of the FSDS form, and verification of the transfer of results from the FSDS forms into the project database. An FSDS review is performed on all samples selected for TEM or PLM data verification.

The data verification review ensures that any data reporting issues are identified and rectified to limit any impact on overall data quality. If issues are identified during the data verification, the frequency of these checks may be increased as appropriate.

Data verification will be performed by appropriate CDM Smith staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements. The data verifier will prepare a data verification report (template reports are included in the SOPs) to summarize any issues identified and necessary corrections. A copy of this report will be provided to the appropriate project data manager, LC, and the EPA RPM. It is the responsibility of the OU3 database manager (CDM Smith) to coordinate with the FTL and/or LC to resolve any OU3 project database corrections and address any recommended field or laboratory procedural changes from the data verifier. The OU3 database manager is also responsible for electronically tracking in the project database which data have been verified, who performed the verification, and when.

9.1.4 Data Validation Method

Unlike data verification, where the goal is to identify and correct data reporting errors, the goal of data validation is to evaluate overall data quality and to assign data qualifiers, as appropriate, to alert data users to any potential data quality issues. Data validation will be performed by the QATS contractor (or their designate), with support from technical support staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements.

Data validation for asbestos should be performed in basic accordance with the *National Functional Guidelines (NFG) for Asbestos Data Review* (EPA 2011d), and should include an assessment of the following:

- Internal and external field audit/surveillance reports
- Field ROMs
- Field QC sample results
- Internal and external laboratory audit reports
- Laboratory contamination monitoring results
- Laboratory ROMs
- Internal laboratory QC analysis results
- Inter-laboratory analysis results
- Performance evaluation results
- Instrument checks and calibration results
- Data verification results (i.e., in the event that the verification effort identifies a larger data quality issue)

A comprehensive data validation effort for OU3 should be completed quarterly and results should be reported as a technical memorandum. This technical memorandum shall detail the validation procedures performed and provide a narrative on the quality assessment for each type of asbestos analysis, including the data qualifiers assigned, and the reason(s) for these qualifiers. The technical memorandum shall detail any deficiencies and required corrective actions.

Electronic files summarizing the records that have been validated, the date they were validated, any recommended data qualifiers and their associated reason codes should be posted to the

OU3 eRoom. It is the responsibility of the OU3 data manager (CDM Smith) to ensure that the appropriate data qualifiers and reason codes recommended by the data validator are added to the project database, and to electronically track in the project database which data have been validated, who performed the validation, and when.

In addition to performing quarterly data validation efforts, it is the responsibility of the QATS contractor to perform a "real-time" evaluation of all blanks, to ensure that any potential contamination issues are quickly identified and resolved. If any blank results are outside the acceptable limits, the QATS contractor should immediately contact the EPA RPM to ensure that appropriate corrective actions are made.

9.2 Reconciliation with User Requirements

Once all samples have been collected and analytical data has been generated, data will be evaluated to determine if study objectives were achieved. It is the responsibility of data users to perform a data usability assessment to ensure that DQOs have been met, and reported investigation results are adequate and appropriate for their intended use. This data usability assessment should utilize results of the data verification and data validation efforts to provide information on overall data quality specific to each investigation.

The data usability assessment should evaluate results with regard to several data usability indicators, including precision, accuracy and bias, representativeness, comparability, completeness, and whether specified analytic requirements (e.g., sensitivity) were achieved. **Table 9-1** provides detailed information for how each of these indicators may be evaluated for the reported asbestos data. The data usability assessment results and conclusions should be included in any investigation-specific data summary reports.

Non-attainment of project requirements may result in additional sample collection or field observations in order to achieve project needs.

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Figures

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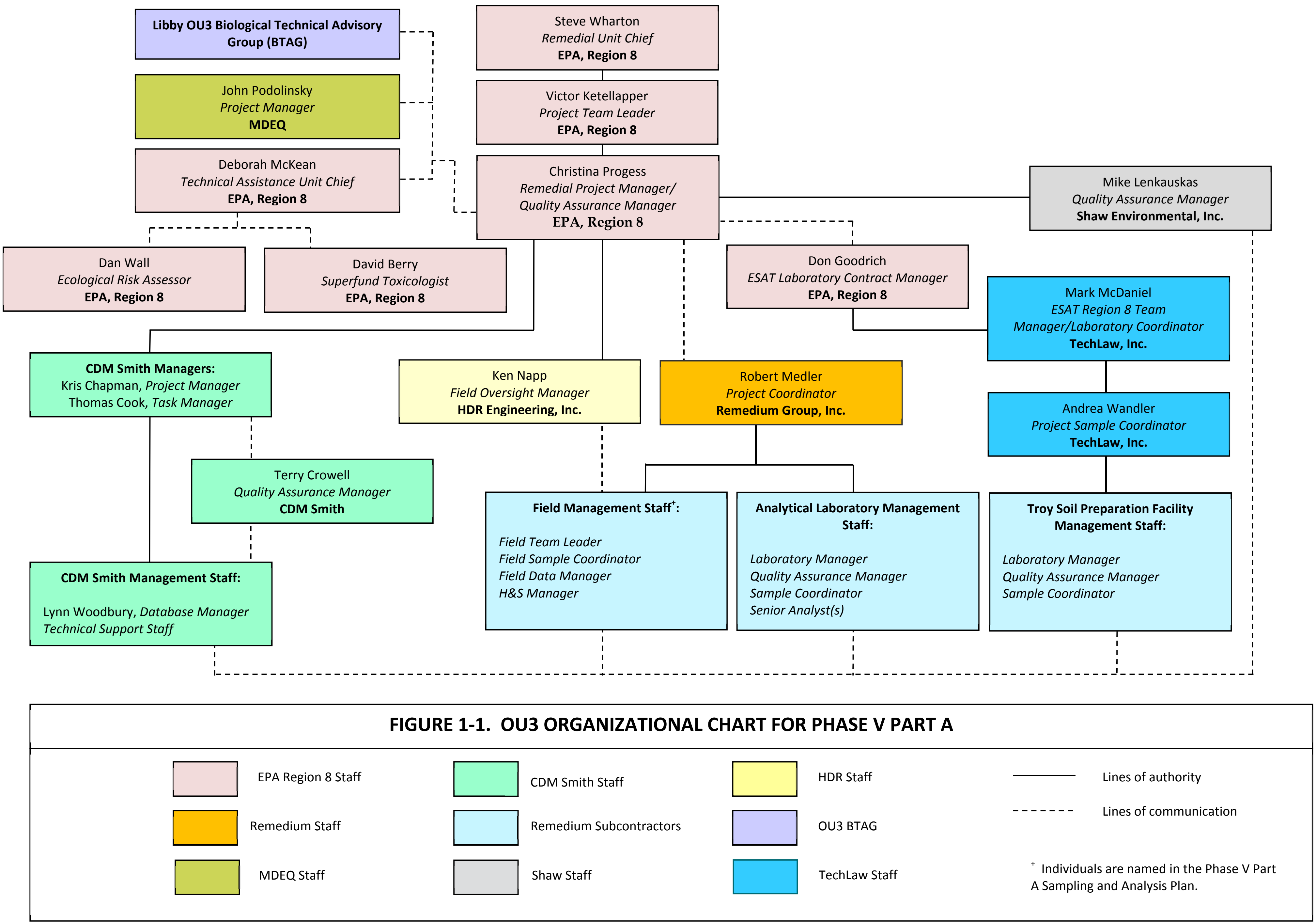

FIGURE 1-1. OU3 ORGANIZATIONAL CHART FOR PHASE V PART A

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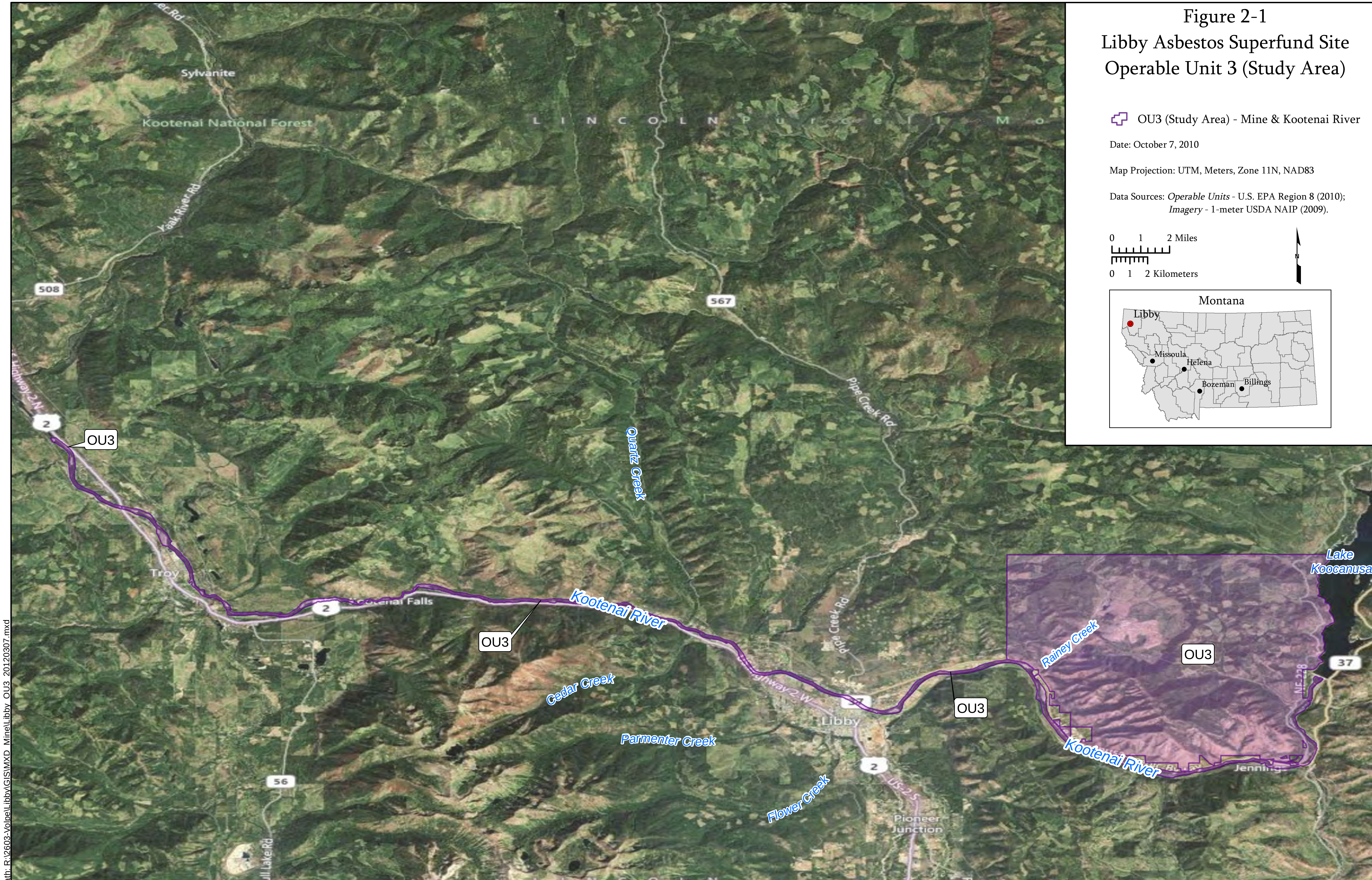

Figure 2-1
Libby Asbestos Superfund Site
Operable Unit 3 (Study Area)

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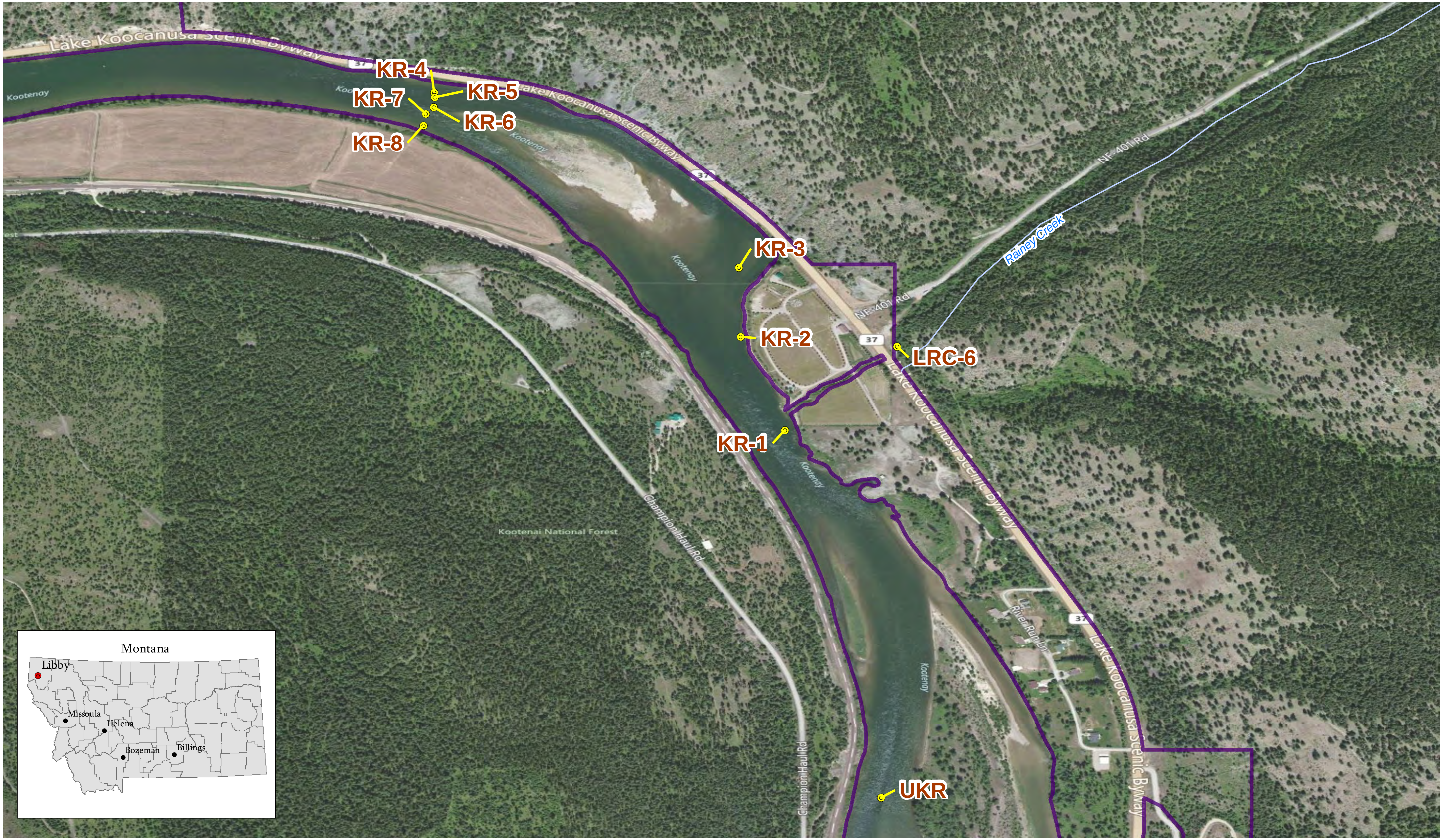

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CDM Smith

Data Sources: Operable Units - U.S. EPA Region 8 (2010); Imagery - Microsoft Bing Maps

N

0 500 1,000 Feet

Key to Features

Surface Water Sampling Locations

OU3 (Study Area) - Mine & Kootenai River

Figure 2-2. Phase IIA Surface Water Sampling Locations Kootenai River

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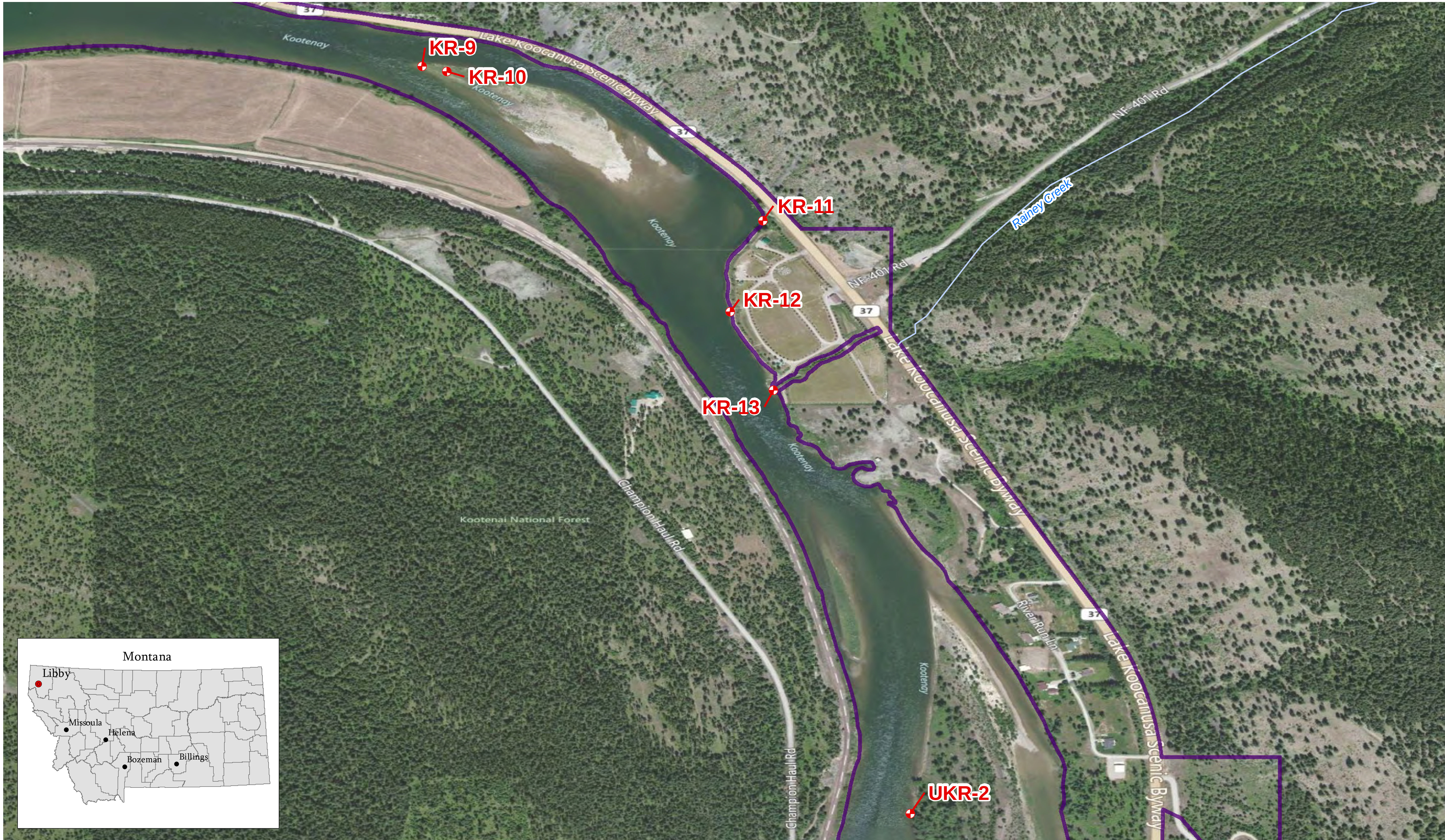

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CDM Smith

Data Sources: Operable Units - U.S. EPA Region 8 (2010); Imagery - Microsoft Bing Maps

N

0 500 1,000 Feet

Key to Features

Sediment Sampling Locations

OU3 (Study Area) - Mine & Kootenai River

Figure 2-3. Phase IIA Sediment Sampling Locations

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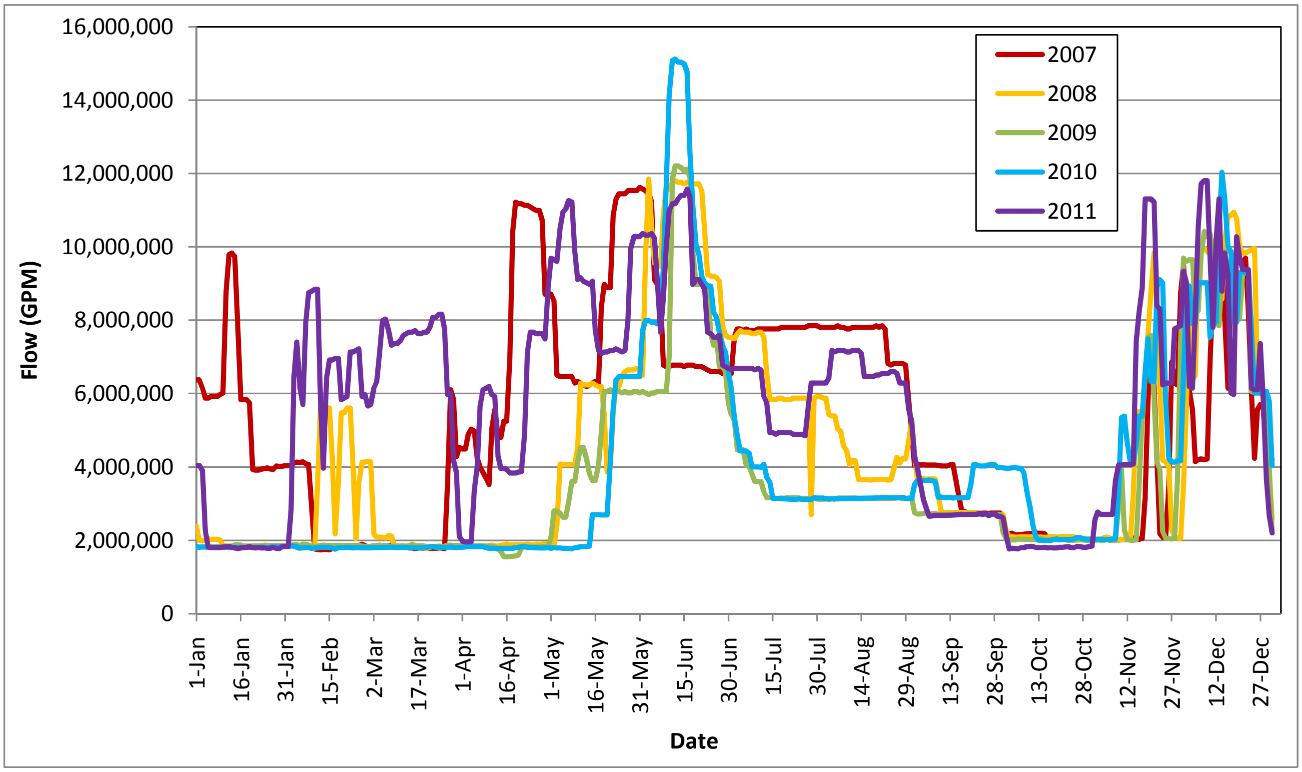
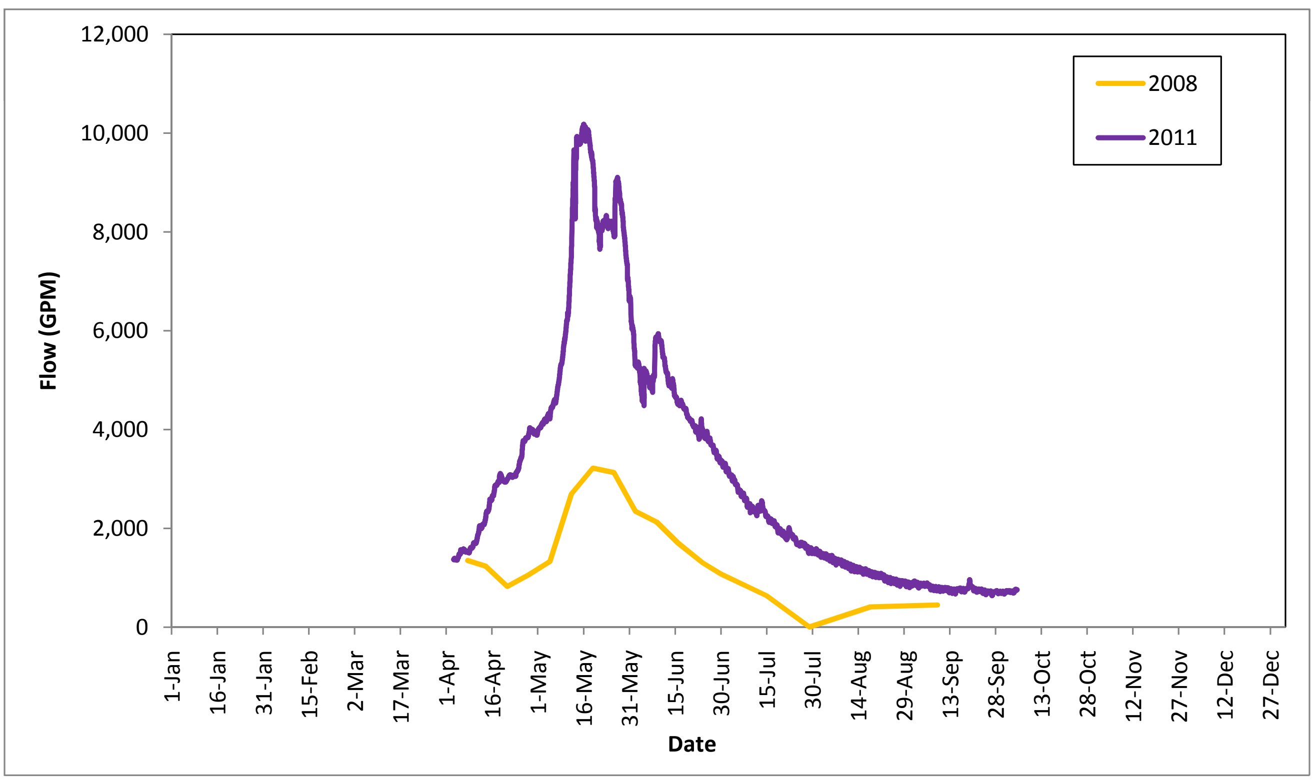
FIGURE 2-4. MEASURED FLOW IN THE KOOTENAI RIVER AND LOWER RAINY CREEK

Panel A. Kootenai River (2007-2011)[a]



Panel B. Lower Rainy Creek Station LRC-6 (2008 and 2011)[b,c]



GPM = gallons per minute

[a] Daily flow measurements for USGS station 12301933.

[b] Flow measurements collected weekly April through June, and biweekly July through September.

[c] Flow measurements collected every half hour from LRC-6.

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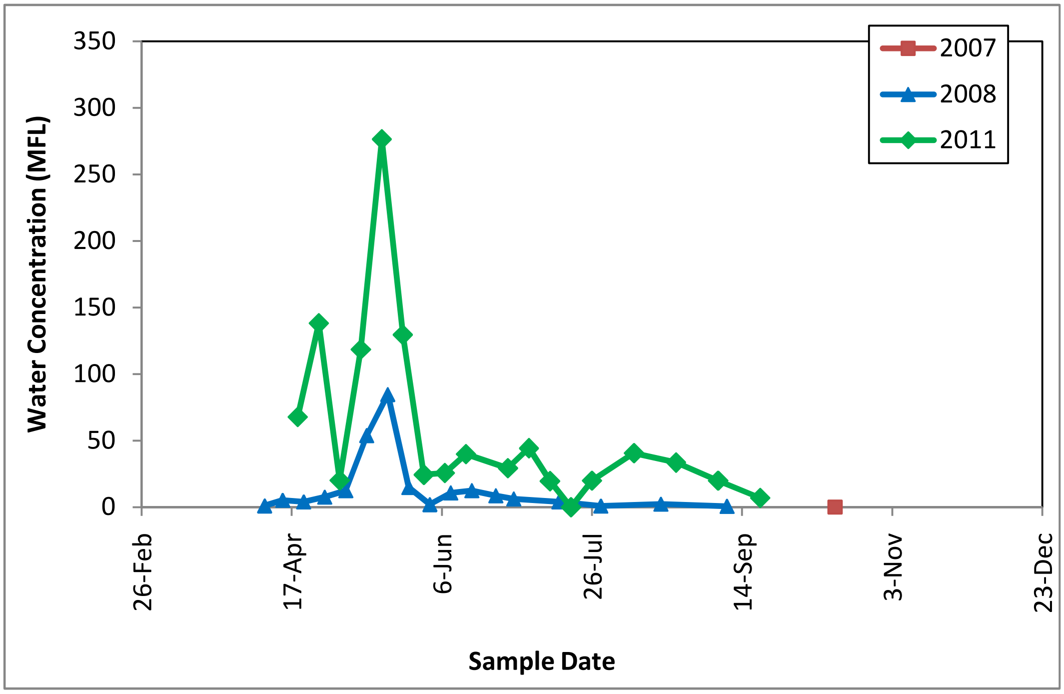
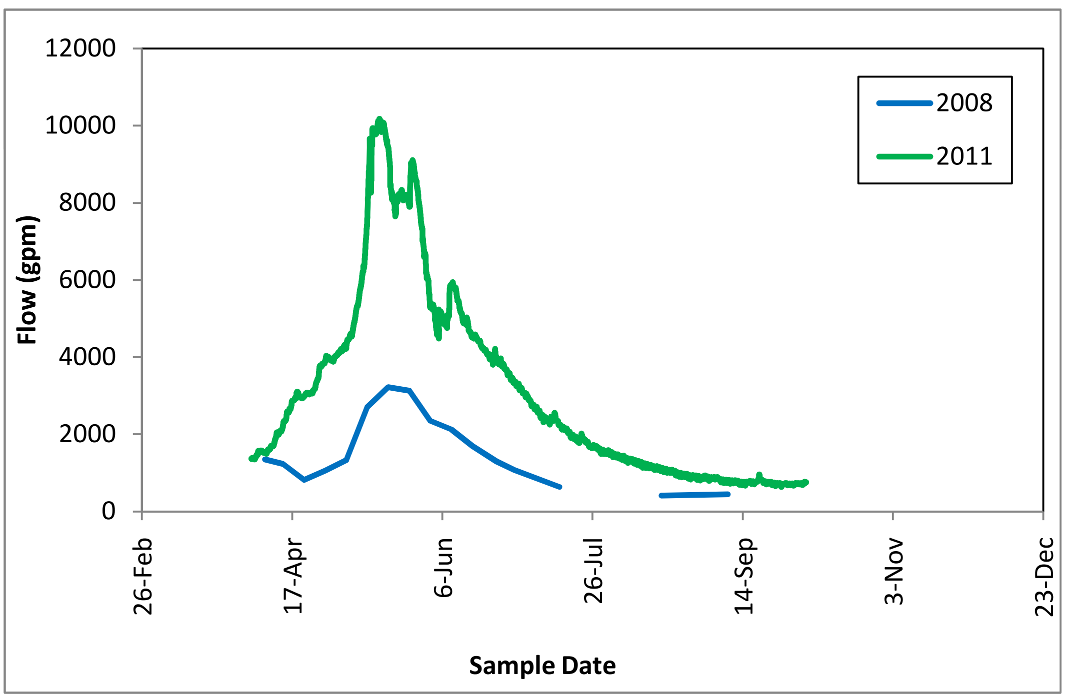
FIGURE 3-1. MEASURED LA CONCENTRATIONS AND FLOW AT LRC-6

Panel A: Total LA Water Concentration



Panel B: Flow



GPM = gallons per minute

MFL = millon fibers per liter

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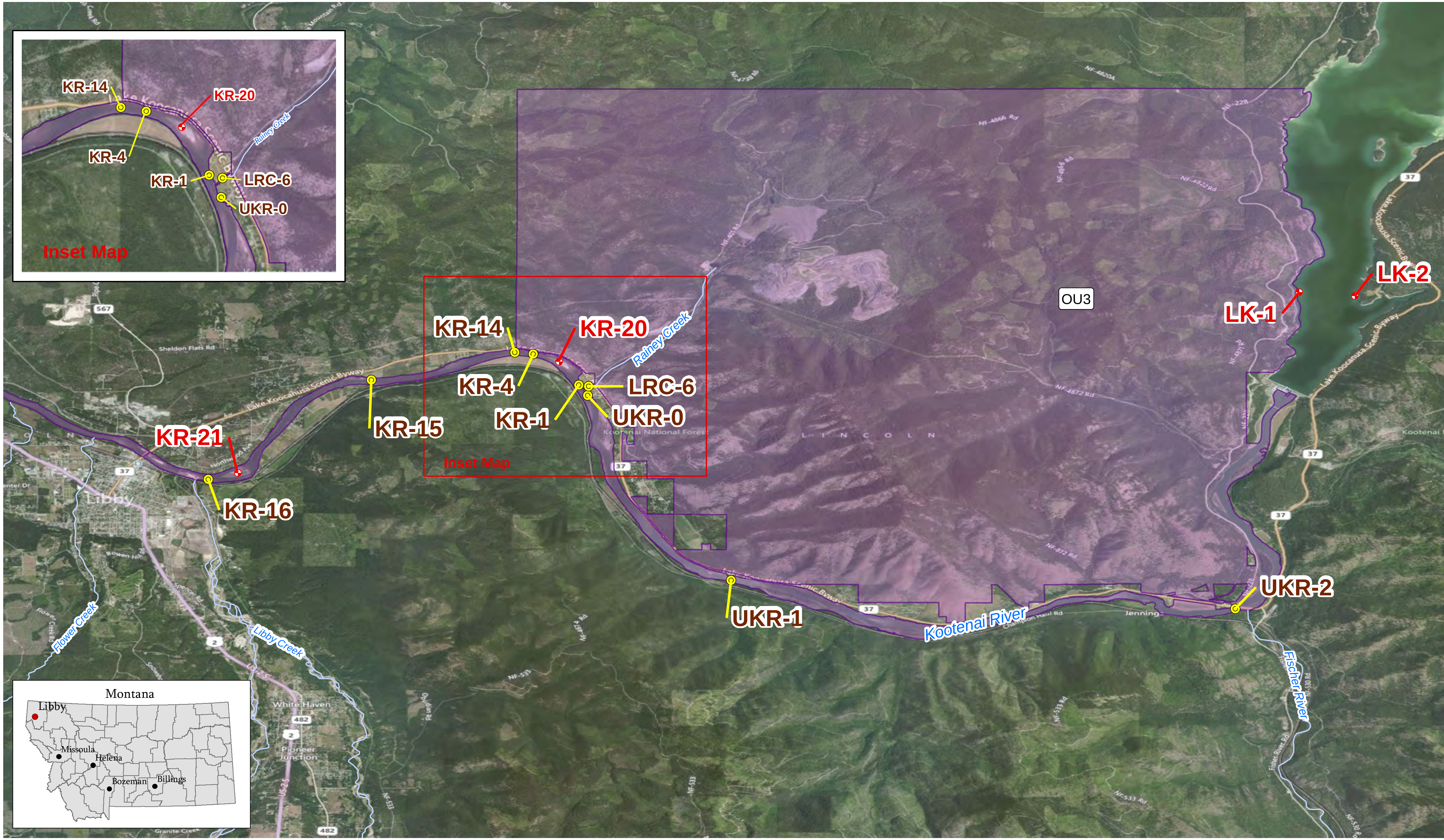

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CDM Smith

Data Sources: Operable Units - U.S. EPA Region 8 (2010); Imagery - Microsoft Bing Maps

N

0 1 2 Miles

0 1 2 Kilometers

Key to Features

Sediment Sampling Station

Surface Water Sampling Stations

OU3 (Study Area) - Mine & Kootenai River

Figure 3.2
Phase VA Sample Locations
Libby Asbestos Superfund Site
Operable Unit 3 (Study Area)

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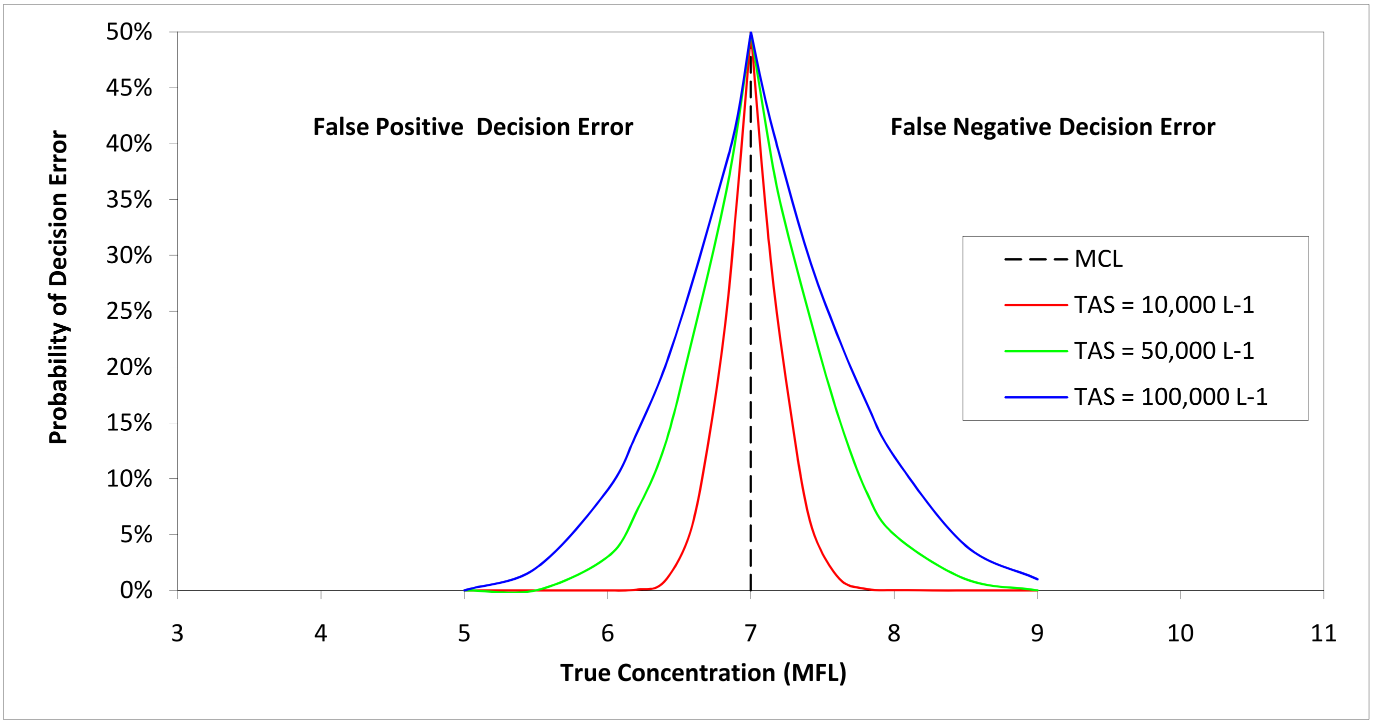
FIGURE 3-3. PROBABILITY OF DECISION ERRORS AS A FUNCTION OF TARGET ANALYTICAL SENSITIVITY



L = liter

MFL = million fibers per liter

TAS = target analytical sensitivity

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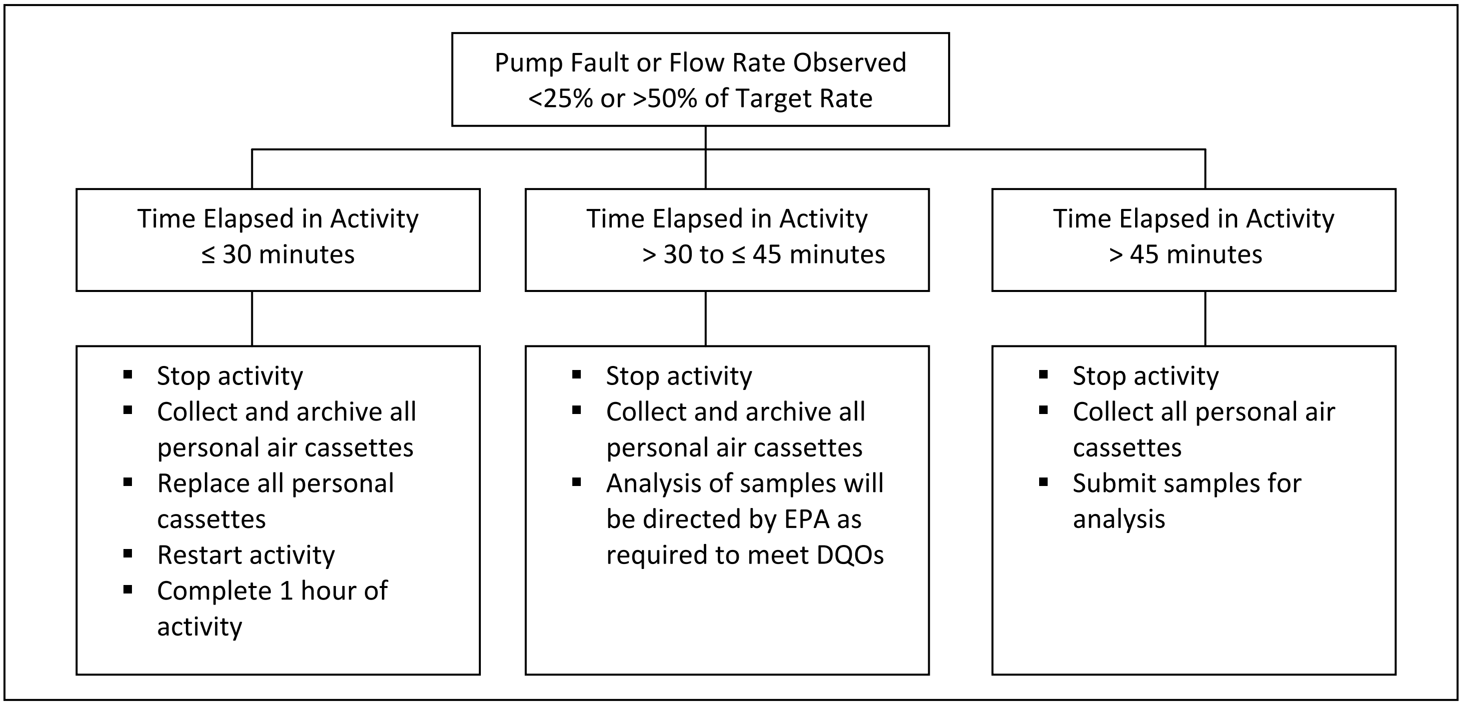
FIGURE 4-1. PROCEDURES FOR PUMP FAULT AND FLOW-RATE ERRORS



Notes: < - less than; > - greater than; ≤ - less than or equal to; % - percent; DQOs – data quality objectives

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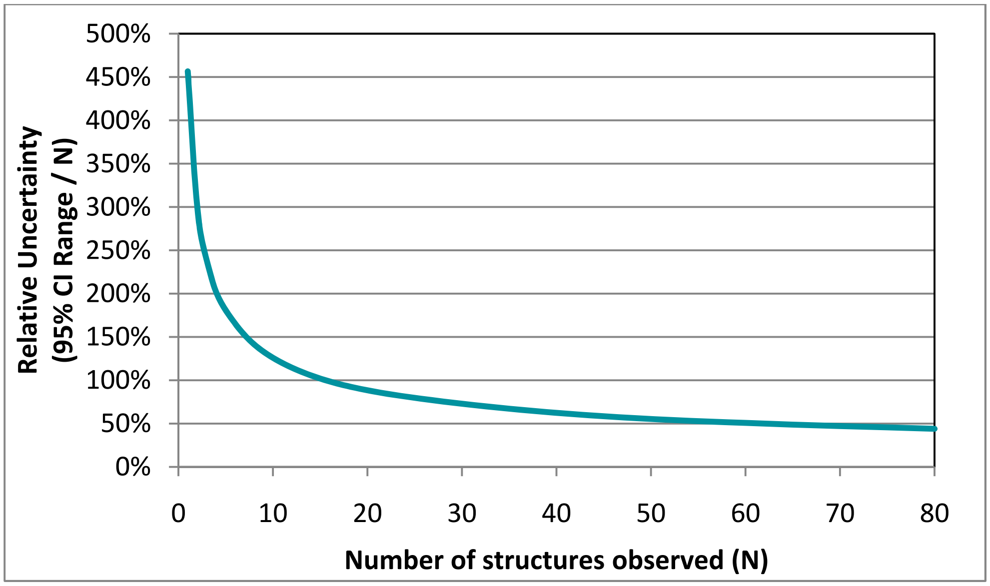
FIGURE 5-1. RELATIONSHIP BETWEEN NUMBER OF STRUCTURES OBSERVED AND RELATIVE UNCERTAINTY



CI = confidence interval

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Tables

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TABLE 1-1. QA/R5 QAPP ELEMENT CROSS-REFERENCE

QA/R-5 QAPP Element	Phase V Part A SAP/QAPP Document Location
Group A. Project Management	
A1. Title & Approval Sheet	Approval Page (pg. 3)
A2. Table of Contents	Table of Contents (pg. 7-10)
A3. Distribution List	Distribution List (pg. 5)
A4. Project/Task Organization	Section 1, Figure 1-1
A5. Problem Definition & Background	Section 2, Section 3.2 to 3.3
A6. Project/Task Description	Section 4, Section 3.2.4, Section 3.3.4
A7. Quality Objectives & Criteria	Section 3.2 to 3.3, Table 9-1
A8. Special Training/Certifications	Field – Section 6.1.1 Analytical Laboratory – Section 6.3.2 to 6.3.4 Troy SPF – Section 6.2.1
A9. Documentation & Records	Field – Section 4.5, Section 4.9.1, Section 6.1.2 Analytical Laboratory – Section 5.2, Section 6.3.5 Troy SPF – Section 5.2, Section 6.2.2
Group B. Data Generation & Acquisition	
B1. Sampling Process Design (Experimental Design)	Section 4.1 to 4.3
B2. Sampling Methods	Section 4.4, Section 4.6
B3. Sample Handling & Custody	Field – Section 4.9 Analytical Laboratory – Section 5.4 Troy SPF – 5.4
B4. Analytical Methods	Section 5.1, Section 5.3, Section 5.5, Appendix G
B5. Quality Control	Field – Section 6.1 Analytical Laboratory – Section 6.3 Troy SPF – Section 6.2
B6. Instrument/Equipment Testing, Inspection, & Maintenance	Field – Section 6.4.1 Analytical Laboratory – Section 6.3.1, Section 6.4.3 Troy SPF – Section 6.4.2
B7. Instrument/Equipment Calibration & Frequency	Field – Section 4.4.2, Section 6.4.1 Analytical Laboratory – Section 6.3.1, Section 6.4.3 Troy SPF – Section 6.4.2
B8. Inspection/Acceptance of Supplies & Consumables	Field – Section 6.5.1 Analytical Laboratory – Section 6.5.2 Troy SPF – Section 6.5.2
B9. Non-direct Measurements	NA
B10. Data Management	Section 7.1 to 7.4
Group C. Assessment & Oversight	
C1. Assessments & Response Actions	Field – Section 8.1.1 Analytical Laboratory – Section 8.1.3 Troy SPF – Section 8.1.2
C2. Reports to Management	Section 8.3, Section 9.1.4
Group D. Data Validation & Usability	
D1. Data Review, Verification, & Validation	Section 9.1
D2. Verification & Validation Methods	Section 9.1.3 to 9.1.4
D3. Reconciliation with User Requirements	Section 9.2

NA – not applicable
QAPP – quality assurance project plan
SAP – sampling and analysis plan
SPF – sample preparation facility

TABLE 2-1. LA RESULTS FOR SURFACE WATER SAMPLES

Location	StationID	Sample Date	Index ID	Sensitivity 1/L	Total LA		LA > 10 µm in Length	
					Count	Conc (MFL)	Count	Conc (MFL)
Upper Kootenai River	UKR	08/19/08	P2-00849	5.0E+04	0	<0.05	0	<0.05
Kootenai River	KR-1	08/19/08	P2-00847	5.0E+04	2	0.10	0	<0.05
	KR-2	08/19/08	P2-00846	5.0E+04	0	<0.05	0	<0.05
	KR-3	08/19/08	P2-00845	5.0E+04	0	<0.05	0	<0.05
	KR-4	08/19/08	P2-00840	5.0E+04	0	<0.05	0	<0.05
	KR-5	08/19/08	P2-00841	5.0E+04	1	0.05	1	0.05
	KR-6	08/19/08	P2-00842	5.0E+04	0	<0.05	0	<0.05
	KR-7	08/19/08	P2-00843	5.0E+04	0	<0.05	0	<0.05
	KR-8	08/19/08	P2-00844	5.0E+04	0	<0.05	0	<0.05

< = less than
> = greater than
µm = microns
Conc = concentration

L = liter
LA = libby amphibole
MFL = million fibers oer liter
mL = milliliters

TABLE 2-2. LA RESULTS FOR SEDIMENT SAMPLES

Location	Station	Sample Date	Index ID	MASS (grams)		RESULTS	
				Fine Fraction	Coarse Fraction	MFLA% Fine	MFLA% Coarse
Upper Kootenai River	UKR-2	8/20/08	P2-00866	123.9	0	ND	--
Kootenai River	KR-9	8/20/08	P2-00860	101	42.9	Tr	ND
	KR-10	8/20/08	P2-00861	82.5	45	Tr	ND
	KR-11	8/20/08	P2-00862	118.5	12.3	Tr	ND
	KR-12	8/20/08	P2-00863	156.7	0	ND	--
	KR-13	8/20/08	P2-00864	116.8	0	Tr	--

-- = not analyzed
ND = non-detect
Tr = trace
MFLA = mass fraction Libby amphibole

TABLE 2-3. NON-ASBESTOS RESULTS FOR LRC-6 SURFACE WATER SAMPLES

Category	Analytical Method	Analyte	Units	Phase I			Phase II					
							Round 1			Round2		
				P1-00300			P2-00401			P2-00880		
				Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier
Metals	SW6020 & SW6010B	Total Aluminum	mg/L	0.09	U		0.11	v		0.09	U	
		Dissolved Aluminum	mg/L	0.09	U		0.09	U		0.09	U	
		Total Antimony	mg/L	0.005	U		0.005	U		0.005	U	
		Dissolved Antimony	mg/L	0.005	U		0.005	U		0.005	U	
		Total Arsenic	mg/L	0.005	U		0.005	U		0.005	U	
		Dissolved Arsenic	mg/L	0.005	U		0.005	U		0.005	U	
		Total Barium	mg/L	0.4	v		0.3	v		0.3	v	
		Dissolved Barium	mg/L	0.4	v		0.3	v		0.4	v	
		Total Beryllium	mg/L	0.0005	U		0.0005	U		0.0005	U	
		Dissolved Beryllium	mg/L	0.0005	U		0.0005	U		0.0005	U	
		Total Cadmium	mg/L	0.0001	U		0.0001	U		0.0001	U	
		Dissolved Cadmium	mg/L	0.0001	U		0.0001	U		0.0001	U	
		Total Chromium	mg/L	0.01	U		0.01	U		0.01	U	
		Dissolved Chromium	mg/L	0.01	U		0.01	U		0.01	U	
		Total Cobalt	mg/L	0.01	U		0.01	U		0.01	U	
		Dissolved Cobalt	mg/L	0.01	U		0.01	U		0.01	U	
		Total Copper	mg/L	0.002	U		0.002	U		0.002	U	
		Dissolved Copper	mg/L	0.002	U		0.002	U		0.002	U	
		Total Lead	mg/L	0.0005	U		0.0005	U		0.0005	U	
		Dissolved Lead	mg/L	0.0005	U		0.0005	U		0.0005	U	
		Total Manganese	mg/L	0.02	U		0.03	v		0.02	U	
		Dissolved Manganese	mg/L	0.02	U		0.02	U		0.02	U	
		Total Nickel	mg/L	0.005	U		0.005	U		0.005	U	
		Dissolved Nickel	mg/L	0.005	U		0.005	U		0.005	U	
		Total Selenium	mg/L	0.005	U		0.005	U		0.005	U	
		Dissolved Selenium	mg/L	0.005	U		0.005	U		0.005	U	
		Total Silver	mg/L	0.001	U	UJ	0.001	U		0.001	U	
		Dissolved Silver	mg/L	0.001	U	UJ	0.001	U		0.001	U	
		Total Thallium	mg/L	0.1	U		0.1	U		0.1	U	
		Dissolved Thallium	mg/L	0.1	U		0.1	U		0.1	U	
		Total Vanadium	mg/L	0.01	U		0.01	U		0.01	U	
		Dissolved Vanadium	mg/L	0.01	U		0.01	U		0.01	U	
		Total Boron	mg/L	0.01	U		0.2	U		0.01	U	
		Dissolved Boron	mg/L	0.01	U		0.03	U		0.01	U	
		Total Calcium	mg/L	79	v		76	v		77	v	
		Dissolved Calcium	mg/L	85	v		70	v		77	v	
		Total Iron	mg/L	0.03	v		0.16	v		0.03	U	
		Dissolved Iron	mg/L	0.03	U		0.03	U		0.03	U	
		Total Magnesium	mg/L	17	v		18	v		18	v	
		Dissolved Magnesium	mg/L	19	v		16	v		18	v	
		Total Potassium	mg/L	9	v		10	v		9	v	
		Dissolved Potassium	mg/L	9	v		9	v		9	v	
		Total Sodium	mg/L	6	v		7	v		6	v	
		Dissolved Sodium	mg/L	7	v		5	v		6	v	
		Total Zinc	mg/L	0.01	U		0.01	U		0.01	U	
		Dissolved Zinc	mg/L	0.01	U		0.01	U		0.01	U	
	SW7470A	Total Mercury	mg/L	0.0006	U	UJ	0.0006	U		0.0006	U	
		Dissolved Mercury	mg/L	0.0006	U	UJ	0.0006	U		0.0006	U	
Extractable Hydrocarbons	SW8015M	Total Extractable Hydrocarbons	ug/L	0.3	U		300	U		300	U	UJ
Volatile Hydrocarbons	MA-VPH	Benzene	ug/L	0.5	U		0.5	U		0.5	U	
		C5 to C8 Aliphatics	ug/L	20	U		20	U		20	U	
		C9 to C10 Aromatics	ug/L	20	U		20	U		20	U	
		C9 to C12 Aliphatics	ug/L	20	U		20	U		20	U	
		Ethylbenzene	ug/L	0.5	U		0.5	U		0.5	U	
		m+p-Xylenes	ug/L	0.5	U		0.5	U		0.5	U	
		Methyl tert-butyl ether (MTBE)	ug/L	1	U		1	U		1	U	
		Naphthalene	ug/L	1	U		1	U		1	U	
		o-Xylene	ug/L	0.5	U		0.5	U		0.5	U	
		Toluene	ug/L	0.5	U		0.5	U		0.5	U	
		Total Purgeable Hydrocarbons	ug/L	20	U		20	U		20	U	
		Xylenes, Total	ug/L	0.5	U		0.5	U		0.5	U	
Nitrogen Compounds	E350.1	Nitrogen, Ammonia as N	mg/L	0.1	U		0.1	U		0.1	U	UJ
	E351.2	Nitrogen, Kjeldahl, Total as N	mg/L	0.5	U		0.5	U		0.5	U	
	E353.2	Nitrogen, Nitrate+Nitrite as N	mg/L	0.02	v	U	0.05	v		0.09	v	
		Nitrogen, Nitrite as N	mg/L	0.01	U		0.01	U	UJ	0.05	U	
	Calculation	Nitrogen, Nitrate as N	mg/L	0.02	v	U	0.05	v		0.09	v	
Anions	E300.0	Chloride	mg/L	6	v		4	v		6	v	
		Fluoride	mg/L	0.6	v		0.9	v		0.7	v	
		Sulfate	mg/L	11	v		10	v		11	v	
	E365.1	Phosphorus, Orthophosphate as P	mg/L	0.132	v		0.179	v	J	0.138	v	J
Water Quality Parameters	A2320 B	Alkalinity, Total as CaCO3	mg/L	279	v		263	v	J	253	v	J
		Bicarbonate as HCO3	mg/L	339	v		302	v	J	303	v	J
		Carbonate as CO3	mg/L	4	U		9	v	J	4	U	
	A2340 B	Hardness as CaCO3	mg/L	290	v		263	v		267	v	
	A2540 C	Solids, Total Dissolved TDS @ 180 C	mg/L	332	v		313	v		309	v	
	A2540 D	Solids, Total Suspended TSS @ 105 C	mg/L	10	U		10	U		10	U	
	A5310 C	Organic Carbon, Dissolved (DOC)	mg/L	NA	NA	NA	3.2	v		2.4	v	

C = Celsius
J = estimated value
mg/L = milligrams per liter
NA = not analyzed
R = rejected
U = non-detect
ug/L = micrograms per liter
v = detect

TABLE 2-4. NON-ASBESTOS RESULTS FOR LRC-6 SEDIMENT SAMPLES

Category	Analytical Method	Analyte	Units	Phase I			Phase II					
							Round 1			Round 2		
				P1-00327			P2-00461			P2-00941		
				Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier
Metals	SW6020 & SW6010B	Aluminum	mg/kg-dry	11200	v		8290	v		11800	v	
		Antimony	mg/kg-dry	0.3	U	UJ	2	U	UJ	2	U	UJ
		Arsenic	mg/kg-dry	2	U		2	U		2	U	
		Barium	mg/kg-dry	855	v		686	v		799	v	J
		Beryllium	mg/kg-dry	5	U		5	U		5	U	
		Boron	mg/kg-dry	5	U		5	U		5	U	
		Cadmium	mg/kg-dry	0.4	U		1	U		1	U	
		Chromium	mg/kg-dry	126	v	J	87	v		136	v	J
		Cobalt	mg/kg-dry	19	v		11	v		17	v	
		Copper	mg/kg-dry	36	v		26	v		28	v	
		Iron	mg/kg-dry	33200	v		15300	v		21200	v	
		Lead	mg/kg-dry	23	v		17	v		18	v	
		Manganese	mg/kg-dry	492	v	J	406	v		524	v	J
		Nickel	mg/kg-dry	31	v		21	v		35	v	
		Selenium	mg/kg-dry	0.5	U		5	U	UJ	5	U	UJ
		Silver	mg/kg-dry	2	U		1	U		1	U	
		Thallium	mg/kg-dry	0.6	U		0.6	U		0.6	U	
		Vanadium	mg/kg-dry	80	v		35	v		42	v	J
		Zinc	mg/kg-dry	26	v		22	v		32	v	
	SW7470A	Mercury	mg/kg-dry	0.1	U		0.2	U		0.2	U	
Extractable Hydrocarbons	MA-EPH	C11 to C22 Aromatics	mg/kg-dry	14	U	UJ	NA	NA	NA	NA	NA	NA
		C19 to C36 Aliphatics	mg/kg-dry	14	U	UJ	NA	NA	NA	NA	NA	NA
		C9 to C18 Aliphatics	mg/kg-dry	14	U	UJ	NA	NA	NA	NA	NA	NA
		Total Extractable Hydrocarbons	mg/kg-dry	14	U	UJ	NA	NA	NA	NA	NA	NA
	SW8015M	Total Extractable Hydrocarbons	mg/kg-dry	76	v		199	v		74	v	
Volatile Hydrocarbons	MA-VPH	Benzene	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
		C5 to C8 Aliphatics	mg/kg-dry	1.9	U		3.3	U	UJ	4.5	U	
		C9 to C10 Aromatics	mg/kg-dry	1.9	U		3.3	U	UJ	4.5	U	
		C9 to C12 Aliphatics	mg/kg-dry	1.9	U		3.3	u	J	4.5	U	
		Ethylbenzene	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
		m+p-Xylenes	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
		Methyl tert-butyl ether (MTBE)	mg/kg-dry	0.097	U		0.17	U	UJ	0.22	U	
		Naphthalene	mg/kg-dry	0.097	U		0.17	U	UJ	0.22	U	
		o-Xylene	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
		Toluene	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
		Total Purgeable Hydrocarbons	mg/kg-dry	1.9	U		5.1	v	J	4.5	U	
		Xylenes, Total	mg/kg-dry	0.049	U		0.083	U	UJ	0.11	U	
Anions	E300.0	Fluoride, 1:2	mg/kg-dry	1	U		1	U		1	U	
	E365.1	Phosphorus, Total	mg/kg-dry	3520	v		3310	v		3390	v	
Sediment Quality Parameters	ASAM10-3.2	pH, sat. paste	s.u.	7.4	v		7.7	v		7.2	v	
	A2540 G	Solids, Total	wt%	29	v		65.2	v		68.1	v	
	Leco	Carbon, Organic	wt%	1.15	v		1.69	v		0.86	v	
Polychlorinated Biphenyls (PCBs)	SW8082	Aroclor 1016	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1221	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1232	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1242	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1248	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1254	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1260	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1262	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
		Aroclor 1268	mg/kg-dry	NA	NA	NA	0.026	U		0.025	U	
Pesticides	SW8081A	4,4′-DDD	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		4,4′-DDE	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		4,4′-DDT	mg/kg-dry	0.0024	U	UJ	NA	NA	NA	NA	NA	NA
		Aldrin	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		alpha-BHC	mg/kg-dry	0.0025	U		NA	NA	NA	NA	NA	NA
		alpha-Chlordane	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		beta-BHC	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Chlordane	mg/kg-dry	0.024	U		NA	NA	NA	NA	NA	NA
		delta-BHC	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Dieldrin	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endosulfan I	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endosulfan II	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endosulfan sulfate	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endrin	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endrin aldehyde	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Endrin ketone	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		gamma-BHC (Lindane)	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		gamma-Chlordane	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Heptachlor	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Heptachlor epoxide	mg/kg-dry	0.0024	U		NA	NA	NA	NA	NA	NA
		Isodrin	mg/kg-dry	0.0028	U		NA	NA	NA	NA	NA	NA

TABLE 2-4. NON-ASBESTOS RESULTS FOR LRC-6 SEDIMENT SAMPLES

Category	Analytical Method	Analyte	Units	Phase I			Phase II					
							Round 1			Round 2		
				P1-00327			P2-00461			P2-00941		
				Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier	Result	Qualifier	Validation Qualifier
		Methoxychlor	mg/kg-dry	0.0024	U	UJ	NA	NA	NA	NA	NA	NA
		Toxaphene	mg/kg-dry	0.24	U		NA	NA	NA	NA	NA	NA
	SW8151A	2,4,5-T	mg/kg-dry	0.0056	U		NA	NA	NA	NA	NA	NA
		2,4,5-TP (Silvex)	mg/kg-dry	0.0056	U		NA	NA	NA	NA	NA	NA
		2,4-D	mg/kg-dry	0.028	U		NA	NA	NA	NA	NA	NA
		Dalapon	mg/kg-dry	0.071	U		NA	NA	NA	NA	NA	NA
		Dicamba	mg/kg-dry	0.0071	U		NA	NA	NA	NA	NA	NA
		Dichlorprop	mg/kg-dry	0.028	U		NA	NA	NA	NA	NA	NA
		MCPA	mg/kg-dry	5.6	U		NA	NA	NA	NA	NA	NA
		MCPP	mg/kg-dry	5.6	U		NA	NA	NA	NA	NA	NA
		Pentachlorophenol	mg/kg-dry	0.0028	U		NA	NA	NA	NA	NA	NA
Polycyclic Aromatic Hydrocarbons (PAHs)	SW8270C	2-Methylnaphthalene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Acenaphthene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Acenaphthylene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Anthracene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Benzo(a)anthracene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Benzo(a)pyrene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Benzo(b)fluoranthene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Benzo(g,h,i)perylene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Benzo(k)fluoranthene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Chrysene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Dibenzo(a,h)anthracene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Fluoranthene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Fluorene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Indeno(1,2,3-cd)pyrene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Isophorone	mg/kg-dry	NA			NA	NA	NA	NA	NA	NA
		Naphthalene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Phenanthrene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA
		Pyrene	mg/kg-dry	0.47	U		NA	NA	NA	NA	NA	NA

J = estimated value
mg'kg = milligrams per kilogram
NA = not analyzed

R = rejected
U = non-detect
v = detect

TABLE 3-1. AVERAGE MONTHLY FLOW FOR LRC-6 AND THE KOOTENAI RIVER

Month	2008			2011		
	Kootenai River Flow (cfs) [a]	LRC-6 Flow (cfs) [b]	Flow Ratio (LRC-6/ Kootenai River)	Kootenai River Flow (cfs) [a]	LRC-6 Flow (cfs) [b]	Flow Ratio (LRC-6/ Kootenai River)
April	4,165	2.5	0.06%	11,386	5.8	0.05%
May	12,016	5.8	0.05%	19,861	16.3	0.08%
June	23,067	3.8	0.02%	20,837	10.2	0.05%
July	14,297	1.4	0.01%	12,919	4.9	0.04%
August	9,524	0.9	0.01%	14,784	2.5	0.02%
September	6,628	1.0	0.01%	6,319	1.7	0.03%
October	4,651	NA	NA	4,107	1.6	0.04%

[a] Flow measurements collected daily.

[b] Flow measurements collected weekly April through June, and biweekly July through September.

[c] Flow measurements collected every half hour from LRC-6.

cfs = cubic feet per second

NA = not available

TABLE 4-1. SAMPLE COLLECTION SUMMARY

Media	Sample Location	Station Description	Station ID	Number of Events		Total Number of Samples
				High Flow	Low Flow	
Surface Water	Lower Rainy Creek	Immediately upstream of the confluence with the Kootenai River	LRC-6	8	1	9
	Kootenai River	Immediately downstream of the Fischer River	UKR-2	1	1	2
		Downstream of multiple minor tributaries entering from the south side of the Kootenai River	UKR-1	1	1	2
		Immediately upstream of the confluence with Rainy Creek	UKR-0	8	1	9
		Immediately downstream of the confluence with Rainy Creek	KR-1	8	1	9
		Immediately downstream of the sandbar located below the confluence with Rainy Creek	KR-4	8	1	9
		Downstream of first minor tributary entering from the north side of the Kootenai River	KR-14	1	1	10 [a]
		Downstream of second minor tributary entering from the north side of the Kootenai River	KR-15	1	1	2
		Immediately upstream of the confluence with Libby Creek	KR-16	1	1	10 [a]
Sediment	Kootenai River	Sandbar below confluence with Rainy Creek	KR-20	0	2	2
		Sandbar above confluence with Libby Creek	KR-21	0	1	1
	Lake Koocanusa	Lake Koocanusa - McGillivray campground	LK-1	0	1	1
		Lake Koocanusa - Lake Koocanusa Marina	LK-2	0	1	1
ABS Air	Kootenai River	Sandbar below confluence with Rainy Creek	KR-20	0	4	4

[a] Five samples will be collected per event, one grab sample from the bank of the river and four transect samples will be collected.

TABLE 4-2. PHASE V PART A SURFACE WATER SAMPLING SCHEDULE

Sample Location	StationID	High Flow (April - June)								Low Flow (~Sept.)
		Week 1	**Week 2**	**Week 3**	**Week 4**	**Week 5**	**Week 6**	**Week 7**	**Week 8**	**Week 9**
		4/23/12	4/30/12	5/7/12	5/14/12	5/21/12	5/28/12	6/4/12	6/11/12	TBD
Lower Rainy Creek	LRC-6	X	X	X	X	X	X	X	X	X
Kootenai River	UKR-2								X	X
	UKR-1								X	X
	UKR-0	X	X	X	X	X	X	X	X	X
	KR-1	X	X	X	X	X	X	X	X	X
	KR-4	X	X	X	X	X	X	X	X	X
	KR-14								X	X
	KR-15								X	X
	KR-16								X	X

TABLE 9-1. GENERAL EVALUATION METHODS FOR ASSESSING ASBESTOS DATA USABILITY

Data Usability Indicator	General Evaluation Method
Precision	Sampling – Review results for co-located samples and field duplicates to provide information on variability arising from medium spatial heterogeneity and sampling and analysis methods. Soil Preparation – Review results for preparation duplicates to provide information on variability arising from sample preparation and analysis methods. Analysis – Review results for PLM laboratory duplicates, TEM recounts, and TEM repreparations to provide information on variability arising from analysis methods. Review results for inter-laboratory analyses to provide information on variability and potential bias between laboratories.
Accuracy/Bias	TEM – Calculate the background filter loading rate and use results to assign detect/non-detect in basic accordance with ASTM 6620-00. For air samples, determine the frequency of indirect preparation. PLM – Review results for LA-specific performance evaluation standards to provide information on direction/magnitude of potential bias. Review results for blanks to provide information on potential contamination.
Representativeness	Review relevant field audit report findings and any field/laboratory ROMs for potential data quality issues.
Comparability	Compare the sample collection SOPs, preparation techniques, and analysis methods to previous investigations.
Completeness	Determine the percent of samples that were able to be successfully collected and analyzed in accordance with the investigation-specific SAP requirements (e.g., 99 of 100 samples, 99%).
Sensitivity	TEM – Determine the fraction of all analyses that stopped based on the area examined stopping rule (i.e., did not achieve the target sensitivity).

ASTM = American Society of Testing and Materials
LA = Libby amphibole
PLM = polarized light microscopy
QATS = Quality Assurance Technical Support
ROM = record of modification
SAP = sampling and analysis plan
SOP = standard operating procedure
TEM = transmission electron microscopy

Appendices

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Appendix A
Standard Operating Procedures (SOPs)

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APPENDIX A

STANDARD OPERATING PROCEDURES

Panel A: Field SOPs[a]

SOP ID	SOP Description
OU3 SOP No. 1	Soil Sampling for Non-Volatile Organic Compound Analysis
OU3 SOP No. 3	Surface Water Sampling
OU3 SOP No. 7	Equipment Decontamination
OU3 SOP No. 8	Sample Handling and Shipping
OU3 SOP No. 9	Field Documentation
OU3 SOP No. 11	GPS Data Collection
OU3 SOP No. 12	Investigation Derived Waste (IDW) Management
OU3 SOP No. 14	Automated Water Sampling and Flow Monitoring
CDM-LIBBY-06	Semi-Quantitative Visual Estimation of Vermiculite in Soils
ABS-LIBBY-OU3	Activity-based Sampling for Asbestos

Panel B: Laboratory SOPs[b]

SOP ID	SOP Description
ISSI-LIBBY-01	Soil Sample Preparation
EPA-LIBBY-08	Indirect Preparation of Samples for TEM Analysis
SRC-LIBBY-01	Analysis of Asbestos in Soil by PLM-Grav
SRC-LIBBY-03	Analysis of Asbestos in Soil by PLM-VE
TEM_WATER_Mod1	OU3-specific Modification to ISO 10312 Method Analysis Of Water Samples For Asbestos By TEM

Panel C: Data Verification SOPs[a]

SOP ID	SOP Description
EPA-LIBBY-09	TEM Data Review and Data Entry Verification
EPA-LIBBY-10	PLM Data Review and Data Entry Verification
EPA-LIBBY-11	FSDS Data Review and Data Entry Verification

[a] The most recent versions of all SOPs are provided electronically in the OU3 eRoom (*https://team.cdm.com/eRoom/mt/LibbyOU3*).

[b] The most recent versions of all SOPs are provided electronically in the Libby Lab eRoom (*https://team.cdm.com/eRoom/mt/LibbyLab*).

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Appendix B
Surface Water Recreational Visitor Activity-Based Sampling Script

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LIBBY SUPERFUND SITE OPERABLE UNIT 3
PHASE V-A SAMPLING AND ANALYSIS PLAN

APPENDIX B
Revision 0

SURFACE WATER RECREATIONAL VISITOR ACTIVITY-BASED SAMPLING (ABS) SCRIPT

This document describes the activities to be conducted by individuals performing the ABS scenario described in the Phase V-A Sampling and Analysis Plan (SAP). Details on the number, location, and timing of ABS sampling events are provided in the Phase V-A SAP.

River guides and recreational users sometimes participate in activities such as boat landing/launching and walking along the banks and on the islands in the Kootenai River that could disturb Libby amphibole (LA) in source material (soil and dried stream-side sediment). The following script will be used to simulate exposures during activities that are considered to be representative of fishermen on islands and along the banks of the Kootenai River. The location where this activity will be conducted is specified in the Phase V-A SAP.

This script does not include transportation for samplers or equipment to and from the island (i.e., sampling will not be conducted during arrival or departure from the island due to health and safety concerns). ABS sampling will be performed by a team of two individual samplers. Each sampler will wear sampling pumps as required in the Phase V-A SAP.

The team of two samplers will begin at the upstream end of the ABS location where they will turn on their sampling pumps. This will initiate both the pump and activity time. Both individuals will then begin to shuffle their feet and gently kick the sediment and rock along the bank remaining within 5 feet of each other. This activity will simulate landing a boat at the ABS area and unloading equipment. After 5 minutes, the samplers will walk separately around the ABS area. The majority of the time should be spent walking alone along the banks, crossing through the interior of the ABS area occasionally. This portion of the activity simulates fishermen moving about the ABS area from one fishing hole to another. The walking activity will last for 50 minutes, bringing the elapsed time to 55 minutes. The shuffling and kicking activity will be repeated for the final 5 minutes, but will be performed along the portion of the ABS area that is parallel to the river current. This will simulate loading equipment and launching a boat. After that 5 minutes (a total of 60 minutes), the ABS event ends, and the air sampling pumps are turned off and the air sampling cassettes are capped.

NOTE: In all cases, it is critical that this sampling effort be performed in a way that does not endanger the health or safety of the sampling personnel. If conditions are considered to be potentially unsafe, the sampler should evacuate the area immediately.

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Appendix C
Record of Modification Forms (ROMs)

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FIELD MODIFICATION APPROVAL FORM
LFM-OU3-xx
Libby OU3 Phase V Part A Sampling & Analysis Plan

Requested by: ____ Date: ______________________

Description of Deviation:

____
____
____
____
____
____
____

☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

____
____
____
____
____
____
____

☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

____
____
____
____
____
____

____ ____ ___________________

Christina Progess, EPA RPM Date

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Request for Modification to **Laboratory Activities** **LB-0000XX**

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

All Labs Applicable Forms – copies to: EPA LC, QATS contractor, All Project Labs

Individual Labs Applicable Forms – copies to: EPA LC, QATS contractor, Initiating Lab

Method (circle all applicable):		TEM-AHERA	TEM-ISO 10312	PCM-NIOSH 7400
	EPA/600/R-93/116	ASTM 5755	TEM 100.2	SRC-LIBBY-03
	SRC-LIBBY-01	NIOSH 9002	Other:______________	

Requester:________________________________ Title: ______________________

Company: ________________________________ Date: ______________________

Original Requester: __________________________ Original Request Date: ______________

[only applicable if modification is a revision of an earlier modification]

Description of Modification:

____

Reason for Modification:

____

Potential Implications of this Modification:

____

Laboratory Applicability (circle one): **All** **Individual(s)** ______________________________

This laboratory modification is (circle one): **NEW** **APPENDS to** ___________ **SUPERCEDES** ____________

Duration of Modification (circle one):

Temporary Date(s): ____

Analytical Batch ID: ______________________________________

Temporary Modification Forms – Attach legible copies of approved form with all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: ___________________

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of method when applicable):

____

REFERENCES

Data Quality Indicator (circle one) – Please reference definitions below for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

DATA QUALITY INDICATOR DEFINITIONS:

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely affect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Technical Review: ____ Date: ____________
(Laboratory Manager or designate)

Project Review and Approval: ______________________________________ Date: ____________
(USEPA: Project Manager or designate)

Approved By: ____ Date: ____________
(USEPA: Technical Assistance Unit Chief or designate)

Appendix D
FSDS Forms

The most recent versions of FSDS forms are provided electronically in the OU3 eRoom (https://team.cdm.com/eRoom/mt/LibbyOU3).

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Appendix E
COC Form

The most recent versions of COC forms are provided electronically in the OU3 eRoom (https://team.cdm.com/eRoom/mt/LibbyOU3).

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Appendix F
TEM Water Modification

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FOR USE AT LIBBY OPERABLE UNIT 3 ONLY

LIBBY OU3 MODIFICATION 1 TO ISO 10312 METHOD ANALYSIS OF WATER SAMPLES FOR ASBESTOS BY TEM
Revision 0

Date: May 21, 2009

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	Bonita Lavelle, USEPA RPM	5/22/09
Modification Author	William Brattin, SRC	5/22/09

Revision	Date	Reason for Revision
0	May 21, 2009	--

1.0 PURPOSE

The purpose of this document is to provide modifications to ISO Method 10312 for use at the Libby Superfund Site Operable Unit 3 in the analysis of water samples for Libby Amphibole (LA) by transmission electron microscopy (TEM).

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that water samples provided to the laboratory for analysis are prepared and analyzed in accord with the requirements of this modification. It is also the responsibility of the Laboratory Director to communicate the need for any deviations from the modification to the appropriate U.S. Environmental Protection Agency (USEPA) Region 8 Remedial Project Manager or Regional Chemist.

3.0 EQUIPMENT

Sample Preparation

- Sonication device
- Oxygen tank
- Ozone generator
- Plastic and glass tubing

Sample Filtration

- NVLAP-compliant High Efficiency Particulate Air (HEPA) hood
- Particle-free water
- Forceps
- Disposable 47 mm filter funnels
- Side arm filter flask
- Mixed Cellulose Ester (MCE) filters, 47 mm diameter, 0.2 µm and 5.0 µm pore size
- Storage container for filters

Grid preparation and Analysis by TEM

All equipment needed for TEM grid preparation and analysis by TEM analysis is detailed in ISO 10312.

4.0 MODIFICATION SUMMARY

Samples of water from field sampling or laboratory-based studies are transmitted to a qualified laboratory for analysis of asbestos. At the laboratory, aliquots of water are filtered, and the filters are analyzed by TEM in accord with ISO 10312 as specified in the applicable Sampling and Analysis Plan. All results are expressed in units of million fibers per liter (MFL).

5.0 SAMPLE PREPARATION

The project-specific Sampling and Analysis Plans should specify if and how water samples should be prepared for analysis. In some cases, no preparation is needed other than ensuring the sample is well-mixed before filtration. In other cases, it may be appropriate to use sonication to disperse clumps of fibers that may be present, or to use sonication and ozone treatment combined, as detailed in EPA Method 100.1 Step 6.2, especially in samples where microbial growth or other organic matter may be present.

6.0 FILTER PREPARATION

After sample preparation (if needed), one or more aliquots of water from each sample will be filtered through 47 mm MCE filters with 0.2 μm pores, using a backing filter with pore size of 5 μm. The volume of water filtered should be selected to provide a filter loading of about 100-1000 asbestos structures per mm^2 on the filter.

For water samples in which it is possible to estimate the concentration before analysis (e.g., samples from a laboratory-based toxicity test), the appropriate volume may be estimated as follows:

$$\text{Volume (mL)} = \frac{\text{Target Loading (s/mm}^2\text{)} \cdot \text{Effective Filter Area (mm}^2\text{)}}{\text{Expected Concentration (s/mL)}}$$

For example, assuming an effective filter area of 1295 mm^2, for the analysis of a sample with an expected concentration of 100 MFL (1E+05 s/mL), a loading of about 500 s/mm^2 would be expected after filtration of about 6 mL.

For water samples for which the concentration can not be reasonably estimated before analysis (e.g., most field samples), then it may be necessary to prepare a series of filters, each with a different volume of water. Typically, this will be done by filtering aliquots of 100 mL, 30 mL, and 10 mL of the sample. Select the filter from the dilution series yielding the largest possible application volume which does not result in an overloaded sample (> 2000 structures per mm^2). If the 10 mL aliquot is overloaded, the laboratory shall prepare a dilution of the sample by removing 5 mL of the remaining volume and diluting to 100 mL. From this secondary dilution, prepare a second series of filters using 60 mL, 20 mL, and 6 mL (corresponding to 3.0 mL, 1.0 ml, and 0.3 mL of the original suspension).

7.0 TEM ANALYSIS

Remove a wedge of about ¼ of the sample filter. Prepare at least 4 grids for TEM analysis as detailed in ISO TEM method 10312, also known as ISO 10312:1995(E). Utilize a minimum of 2 grids (typically 3) for analysis, distributing grid openings examined distributed approximately evenly across the grids. Archive the remaining grid(s) and the remaining filter.

Counting rules

All water samples submitted for asbestos analysis by TEM will be analyzed in basic accord with the ISO 10312 counting protocols, with all applicable Libby site-specific Laboratory Modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000066, and LB-000085.

Stopping Rules

The target analytical sensitivity for sample analysis should be specified in the project-specific SAP. In the absence of such specification, the default target analytical sensitivity for asbestos in water is 50,000 f/L (0.05 MFL). Stopping rules for these analyses are as follows:

1. Calculate the number of grid openings (GOs) needed to achieve the target sensitivity.
2. Count a minimum of 2 GOs in each of 2 grids.
3. Continue counting until one of the following stopping rules is achieved:
    a. The target sensitivity is achieved
    b. A total of 50 Libby amphibole (LA) structures have been counted
    c. A total area of 0.5 mm^2 (usually about 50 GOs) has been examined
4. When one of these rules has been achieved, finish counting the final GO, then stop.

Data Recording and Electronic Data Deliverable

Standard Analysis

Unless otherwise specified in the project-specific SAP, all amphibole structures (including not only LA but all other amphibole asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length $\geq$ 0.5 μm and an aspect ratio (length:width) $\geq$ 3:1, will be recorded on the most recent version of the Libby site-specific laboratory bench sheets and EDD spreadsheet ("TEM Water EDD.xls"). Data recording for chrysotile, if observed, is not required.

Rapid Turn-Around Analysis

In some cases, the project-specific SAP may specify that some water samples shall be analyzed using a “rapid turn-around” protocol. The rapid turn-around protocol differs from the standard analysis as follows:

1. Quantitative measurement of length and width is not required for structures that can be readily classified as countable by eye. Measurements may be necessary for structures that are near the size cutoffs for counting (i.e., length close to 0.5 μm, aspect ratio close to 3:1).
2. Recording of individual structure dimensions and characteristics is not required.
3. Electronic documentation of EDS spectra or SAED patterns is not required.
4. Classification of structures in accord with Libby Laboratory Modification #LB-00066 is not required.

The total number of LA structures observed in each grid opening should be recorded on the most recent version of the Libby site-specific laboratory bench sheets and EDD spreadsheets ("Rapid TEM Water EDD.xls").

8.0 QUALITY CONTROL

The project-specific Sampling and Analysis Plan should specify the types and number of laboratory quality control (QC) samples that should be prepared during the project. In the absence of information in the sampling and Analysis Plan, default guidelines for QC samples are provided in Table 1. This table includes default requirements on the frequency that these QC analyses should be performed, how samples will be selected for QC analyses, the acceptance criteria and corrective actions for these analyses. It is the responsibility of the laboratory manager to ensure that QC requirements are met.

9.0 REFERENCES

International Organization for Standardization (ISO). 1995. Ambient Air – Determination of asbestos fibers – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

TABLE 1
LABORATORY QUALITY CONTROL SAMPLE DEFAULT REQUIREMENTS [a]

Lab QC Type & Description	Analysis Frequency [b]	Acceptance Criteria	Corrective Action(s)
Lab Blank A filter that is prepared using laboratory water.	1% (1 per 100 analyses)	No asbestos structures observed in an analysis of 10 GOs.	The laboratory shall immediately investigate the source of the contamination and take steps to eliminate the source of contamination before analysis of any investigative samples may continue.
Repreparation Prepared by applying a second aliquot of sample water to a new filter, which is then prepared and analyzed in the same fashion as the original filter.	2% (1 per 50 analyses) See note [c]	No more than 5% of the original-repreparation pairs are statistically different from each other at the 90% confidence interval. See note [d]	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, etc).
Recounts **Recount Same.** A re-examination the same grid openings as were evaluated in the original analysis by the same microscopist who performed the initial examination. **Recount Different.** A re-examination the same grid openings as were evaluated in the original analysis by a different microscopist within the same laboratory who performed the initial examination.	2% (1 per 50 analyses) See note [c]	See Libby Laboratory Modification LB-000029	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in counting rules, etc).
Interlabs A re-examination the same grid openings as were evaluated in the original analysis by a different laboratory who performed the initial examination.	1% (1 per 100 analyses) See note [e]	See Libby Laboratory Modification LB-000029	A senior laboratory analyst at the interlaboratory will contact the originating laboratory to discuss the basis of the discordance. As appropriate, each laboratory will take appropriate corrective action (e.g., re-training in counting rules, etc).

[a] Unless specified otherwise in the project-specific sampling and analysis plan or quality assurance project plan.
[b] When calculating the number of QC analyses required for a project, round up to the nearest whole number.
[c] To be selected by the laboratory in accord with the procedures in Attachment 1 in Libby Laboratory Modification LB-000029.
[d] See Attachment 4 in Libby Laboratory Modification LB-000029 for details on performing this statistical comparison.
[e] To be selected by EPA (or EPA's technical contractor) in accord with the procedures in Attachment 2 in Libby Laboratory Modification LB-000029.

Appendix G
Analytical Requirements Sheet

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SAP ANALYTICAL SUMMARY # OU3PHVA-0412
SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS

SAP Title: Phase V Sampling and Analysis Plan for Operable Unit 3, Libby Asbestos Superfund Site - Part A: Kootenai River

SAP Date/Revision: April 17, 2012 (Rev. 0)

EPA Technical Advisor: Christina Progess (303-312-6009, progess.christina@epa.gov)
(contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: The purpose of Part A of the Phase V SAP/QAPP for OU3 is to guide the collection of data on LA in surface water and sediment in the Kootenai River to assess the impact of releases from the mined area. In addition, this program will include activity-based sampling of a recreational area on the Kootenai River.

Estimated number and timing of field samples:

Surface Water --
- >> Weekly high-flow sampling (April-June) = 45 samples + field QC samples
- >> One-time low flow sampling (September) = 17 samples + field QC samples

Sediment --
- >> Sediment sampling (September) = 3 samples + field and preparation QC samples
- >> ABS area (August, September) = 2 samples + field and preparation QC samples

ABS Air --
- >> ABS sampling (August, September) = 4 samples + field QC samples

Index ID Prefix: P5-1xxxx

PCM/TEM Preparation and Analytical Requirements for Water Samples [a]:

Medium Code	Medium	Preparation Details [b]				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep? With Ashing	Indirect Prep? Without Ashing	Filter Archive?	Method	Recording Rules	Analytical Sensitivity/ Stopping Rules	
A	Surface Water (high flow)	Yes	No	No	Yes	Rapid turn-around TEM	*See OU3 TEM Method Modification 1 for ISO 10312 Analysis of Water*	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 50,000 L^{-1} is achieved ii) 25 structures are recorded iii) A total filter area of 1.0 mm2	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

Medium Code	Medium	Preparation Details [b]				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep?		Filter Archive?	Method	Recording Rules	Analytical Sensitivity/ Stopping Rules	
			With Ashing	Without Ashing					
								has been examined (approx. 100 grid openings)	
B	Surface Water (subset of high flow, all low flow)	Yes	No	No	Yes	Standard TEM; ISO 10312	All asbestos [c]; L: ≥ 0.5 µm AR: ≥ 3:1	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 50,000 L^{-1} is achieved ii) 25 structures are recorded iii) A total filter area of 1.0 mm^2 has been examined (approx. 100 grid openings)	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

[a] Rapid turn-around TEM analysis will be performed first on a subset of high-flow samples (as indicated on the COC). EPA will provide direction following receipt of the results of the rapid turn-around TEM analysis, as to which high flow samples will undergo standard TEM analysis. All low flow samples will be analyzed by standard TEM methods.
[b] Sample and filter preparation should be performed in basic accordance with EPA Method 100.2 (as modified by LB-000020A). Grid preparation should be performed in basic accordance with Section 9.3 of ISO 10312:1995(E).
[c] If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

PLM Preparation and Analytical Requirements for Sediment Samples:

Medium Code	Medium	Preparation Method[d]	Analysis Method	Applicable Laboratory Modifications
B	Sediment	ISSI-LIBBY-01 Rev. 11	PLM-Grav: SRC-LIBBY-01 Rev. 3 PLM-VE: SRC-LIBBY-03 Rev. 3	N/A

[d] Sample preparation to be performed at the Troy sample preparation facility and shipped to the PLM analytical laboratory.

TEM Preparation and Analytical Requirements for Air Field Samples:

Medium Code	Medium, Sample Type	Preparation Details				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep?		Filter Archive?	Method	Recording Rules	Analytical Sensitivity/Prioritized Stopping Rules	
			With Ashing	Without Ashing					
C	Air, ABS	*All samples will be archived for future analysis; analytical requirements will be specified at a later date.*							

TEM Preparation and Analytical Requirements for Air Field Quality Control Samples:

Medium Code	Medium, Sample Type	Preparation Details			Analysis Details			Applicable Laboratory Modifications (current version of)
		Indirect Prep?		Archive?	Method	Recording Rules	Stopping Rules	
		With Ashing	Without Ashing					
D	Air, lot blank	No	No	Yes	TEM – ISO 10312	All asbestos; L: $\geq$ 0.5 µm AR: $\geq$ 3:1	Examine 10 grid openings.	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

Laboratory Quality Control Sample Frequencies:

TEM [e]: Lab Blank – 4%
Recount Same – 1%
Verified Analysis – 1%
Repreparation – 4%
Recount Different – 2.5%
Inter-laboratory - 2% [f]

[e] See LB-000029 for selection procedure and QC acceptance criteria.
[f] *Post hoc* selection to be performed by the QATS contractor.

PLM [g]: Lab Duplicates – 10% (cross-check 8%; self-check 2%)
Inter-laboratory – 1% [h]

[g] See SRC-LIBBY-03 for selection procedure and QC acceptance criteria.
[h] *Post hoc* selection to be performed by the QATS contractor.

Asbestos Analytical Laboratory Review Sign-off:

☐ EMSL – Libby [sign & date:_________________________]
☐ EMSL – Cinnaminson [sign & date:_____________________]
☐ EMSL – Beltsville [sign & date:_______________________]
☐ EMSL – Denver [sign & date:_______________________]
☐ ESAT [sign & date: ___________________________]
☐ Hygeia [sign & date:___________________________]

[Checking the box and signing (electronically) above indicates that the laboratory has reviewed and acknowledged the preparation and analytical requirements associated with the specified SAP.]

Requirements Revision:

Revision #:	Effective Date:	Revision Description
0	4/17/12	--

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Appendix H
Pilot Study:
Evaluation of Potential Fiber Loss
While Sampling Water with a Peristaltic Pump

APPENDIX H

PILOT STUDY:
EVALUATION OF POTENTIAL FIBER LOSS
WHILE SAMPLING WATER WITH A PERISTALTIC PUMP

1.0 INTRODUCTION

As discussed in the Phase V Part A Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP) for Libby OU3 (CDM Smith 2012), some samples of surface water are currently planned for collection using a peristaltic pump. Because sampling with a peristaltic pump involves passing water through a plastic tube, EPA has determined that it is appropriate to confirm that this type of sampling does not result in a loss of LA fibers due to potential fiber binding to the tubing.

This pilot study is an Addendum to the Phase V Part A SAP/QAPP, and is designed to test whether water samples collected using a peristaltic pump may tend to underestimate true LA concentrations due to binding of LA to the peristaltic tubing.

2.0 STUDY DESIGN

a) Obtain six clean 1-liter high density polyethylene (HDPE) bottles. Mark each bottle on the outside at the 400 milliliter (mL) and 800 mL levels with an indelible marker.
b) Travel to station LRC-6 on Lower Rainy Creek. Fill each bottle to approximately the 800 mL mark in basic accordance with the direct sampling methods described in OU3 SOP No. 3, except that the open bottle should be filled by holding the bottle horizontally in the upper portion of the flowing stream, with the neck pointed upstream. Use care to avoid disturbance or inclusion of sediment in the water samples.
c) Using a peristaltic pump, pump approximately 400 mL from each bottle into a second clean 1-liter HDPE bottle.
d) Label the 3 bottles filled directly from the stream as:
   - LRC6-D1
   - LRC6-D2
   - LRC6-D3

   Label the 3 bottles filled with a peristaltic pump as:
   - LRC6-P1
   - LRC6-P2
   - LRC6-P3
e) Transport all six bottles to the EMSL analytical laboratory in Libby.
f) The laboratory shall prepare each bottle for analysis using ozone/UV treatment in basic accordance with the techniques in EPA Method 100.2, as modified by Libby Laboratory Modification LB-000020A.

g) Filter equal volumes of ozonated sample through 25-mm diameter polycarbonate filters with a pore size of 0.1 µm with a mixed cellulose ester filter (0.45 µm pore size) used as a support filter.

h) Prepare a minimum of three grids from each filter using the grid preparation techniques described in Section 9.3 of International Organization for Standardization (ISO) 10312:1995(E) (ISO 1995).

i) Examine the grids by TEM in basic accordance with the procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085.

j) Count all structures with fibrous morphology, an x-ray diffraction pattern consistent with amphibole asbestos, a energy dispersive spectrum consistent with LA, length greater than or equal to 0.5 µm, and an aspect ratio (length:width) greater than or equal to 3:1. Raw structure data (i.e., structure type, length, width, etc.) will be recorded on the OU3-specific bench sheets and electronic data deliverable (EDD) spreadsheet for TEM analyses of water.

k) TEM Stopping rules are as follows:
    1. Count a minimum of two grid openings from each of two grids.
    2. Continue counting until one of the following is achieved:
        a. A target analytical sensitivity of 50,000 L^{-1} has been achieved.
        b. 25 LA structures have been observed.
        c. A total filter area of 1.0 mm^2 has been examined

   When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

l) Promptly report all results to EPA (Christina Progess) and the OU3 project data manager (CDM Smith) for evaluation. All TEM EDDs should also be posted to the OU3 eRoom in the appropriate folder.

m) Data Analysis:
    - Compare the matched pairs of direct and pumped samples using the Poisson ratio test.
    - Compare the mean concentration for the three direct-filled samples to the mean of the three pumped samples.
    - If the pumped samples are not statistically lower than the direct-filled samples, and if the mean of the pumped samples is not meaningfully lower (e.g., > 25%) than the mean of the direct-filled samples, conclude that sample by peristaltic pumping is acceptable. If one or more of the pumped samples is statistically lower than the matched direct-filled sample, or if the mean of the pumped samples is meaningfully lower (e.g., > 25%) than the direct-filled sample, conclude that pumping may tend to bias samples low, and do not use unless peristaltic sampling further evaluations indicates otherwise.

3.0 REFERENCES

CDM Smith. 2012. Sampling and Analysis Plan/Quality Assurance Project Plan for Operable Unit 3, Libby Asbestos Superfund Site. Phase V, Part A: Kootenai River Surface Water, Sediment, and Activity-Based Sampling. Prepared by CDM Smith for and with oversight by U.S. EPA, Region 8. April 2012.

OU3 SOP No. 3, Surface Water Sampling (available in the OU3 eRoom), available online at https://team.cdm.com/eRoom/mt/LibbyOU3